UPGRADING AND EXTENDED TESTING

OF THE MSC INTEGRATED WATER

AND WASTE MANAGEMENT HARDWARE

May, 1972
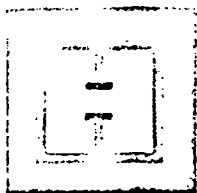
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UPGRADING AND EXTENDED TESTING

OF THE MSC INTEGRATED WATER

AND WASTE MANAGEMENT HARDWARE

(Contract NAS9-9191)

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FOREWORD

This report summarizes the results of the work performed by the AMGLO Corporation and CHEMTRIC Incorporated under Contract NAS9-9191 - for upgrading and extended testing of the Manned Spacecraft Center (MSC) Integrated Water and Waste Management Hardware. This program was sponsored by and performed for the Crew Systems Division of the NASA MSC. Mr. M. L. Owen (EC3) was the designated Technical Monitor.

The work reported herein was started by the AMGLO Corporation in February 1969, and completed by CHEMTRIC Incorporated in December 1971. Contract NAS9-9191, and the personnel and equipment assigned to this program, were transferred to CHEMTRIC (an AMGLO subsidiary) on 1 January 1970. This transfer was authorized by a Novation Agreement.

Robert A. Bambenek (Program Manager), Phillip P. Nuccio (Project Engineer), Thomas L. Hurley (Project Biochemist), and Walter J. Jasionowski (Chemical Engineer) submitted the draft copy of this report in March 1972. Other personnel that made substantial contributions to this program are: Frank Chybik, Jr. (Electronics Specialist), Mahendrasinh D. Rana (Design Engineer), Muganlal Patel (Test Engineer), Peter W. Glocker (Designer), and Andrew L. Murman (Technician). The authors are indebted to these individuals - and Charles Verostko of the NASA MSC for his assistance in obtaining detail analyses of water and waste samples.

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ABSTRACT

This report summarizes the results of three years of work on Contract NAS9-9191 for upgrading and testing an integrated water and waste management system - which uses the compression distillation, reverse osmosis, adsorption filtration and ion-exchange processes to recover potable water from urine, flush water and used wash water. More than 2000 hours of testing was performed with the distillation unit to determine it's performance under a wide variety of conditions - and determine the operational procedures which maximize water yield and quality, and minimize the demand for electric power and expendables. This work also included the development of techniques for (1) extending the useful biological life of biological filters, activated carbon filters and ion-exchange resins to at least 30 days, and (2) presterilizing ion-exchange resins so that sterile water can be recovered from waste water.

A wide variety of reverse osmosis materials, surfactants and germicides were experimentally evaluated to determine the best combination for a wash water subsystem. Full-scale module tests with real wash water demonstrated that surface fouling is a major problem. The two most promising modules are recommended for additional testing under conditions which will reduce their effective weight penalties. Minor changes in the design of the distillation unit are recommended before the development of a flight-qualified system is undertaken.

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INTRODUCTION & SUMMARY

1.1 Background

Manned spacecraft designed to accomplish long duration missions will contain equipment for recovering water from metabolic wastes - to circumvent the untenable weight and volume requirements associated with carrying fresh water and disposing of used water. To assure the availability of an effective water recovery system design for these missions, the NASA MSC has supported the development of prototype water and waste management hardware. This report summarizes the development work performed under contract NAS9-9191 on an integrated system which utilizes compression distillation, reverse osmosis, adsorption filtration and ion-exchange processes to recover potable water from urine, flush water and used wash water.

The initial MSC funded efforts on the development of an integrated water and waste management were conducted by The Marquardt Corporation. They developed a compression distillation unit under NAS9-1680, and a prototype model of an integrated water and waste management system under NAS9-5119. These efforts resulted in the development of hardware which has the desired operational potential; however, subsequent tests indicated the need for additional work to confirm the suitability of this design for long term missions.

The hardware developed by Marquardt under NAS9-5119 was transferred to AMGLO for refurbishment and testing under contract NAS9-9014. The results of this work, as described in AMGLO Final Report 3080 (October 1969), are summarized as follows.

A. The hardware as received from the NASA MSC was in reasonably good condition; however, many components were rearranged, modified and/or eliminated to improve reliability and performance.

B. Calibration tests with distilled water indicated that the performance of the two distillation units contained within the system is substantially less than predicted by Marquardt. From the data obtained, it was concluded that (1) the diaphragm-type compressors used have a volumetric efficiency less than 60% instead of near 100%, and (2) because of inboard leakage through dynamic seals the water yield is less than 82% instead of more than 98%. Also, it was concluded that solids can not be dried inside the distillation units because of their thermal design.

C. A 30-day, around-the-clock test was then performed to evaluate the quality of water recovered from urine and fecal slurries. During this test the distillation units

experienced frequent mechanical failures - and the feed line in the fecal still was frequently plugged with fecal solids. The quality of the raw condensate was poor in comparison to the water recovered by previously developed compression distillation units; however, after charcoal filtration, dosing with AgCl ions, and ion-exchange, it's quality exceeded current standards for potability - and no coliform bacteria were detected. In addition, this test demonstrated that the use of a biological filter upstream of the charcoal filter extends the useful life of this filter from 3 to 10 days. Also, AgCl-dosing maintained sterile conditions in the condensate storage tanks - but not in the waste storage tank nor in the fecal collector.

From these results it was concluded that an improved water and waste management system should:

A. Use a chemical disinfectant in the urinal and commode flush water.

B. Filter-out any suspended solids contained in the feed to the distillation units.

C. Use a distillation unit which (1) operates at saturation temperatures near cabin temperature instead of 120°F, (2) contains no more than one dynamic seal, and (3) pumps concentrated waste water to an external dryer.

D. Utilize pasteurization temperatures to sterilize the ion-exchange resin, and/or stored potable water.

All of these improvements, plus others, were made under contract NAS9-9191.

1.2 Objectives

The primary objective of contract NAS9-9191 was to upgrade the hardware tested under contract NAS9-9014 - to furnish an integrated system for recovery of water from all onboard sources and thus, close the water cycle. Originally this was to be accomplished by adding a wash water recovery subsystem. However, after the NAS9-9014 results were obtained the objectives were modified to include the development of a new compression distillation unit which includes an external solids dryer. The program requirements then were defined as follows.

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Par 4.1* Perform the analyses required to establish the design requirements for integrating a wash water recovery system with the current hardware (i.e., NAS9-9014).

Par 4.2 Conduct experiments with available reverse osmosis membranes, surfactants and germicides - to define membrane life, membrane-surfactant-germicide compatability, feed and flow control, and operating pressure requirements. Also, define the requirements for a wick-type solids dryer by (1) screening candidate wicking materials, and (2) performing simulated 30-day operational cycles.

Par 4.3 Prepare a preliminary design for adding a wash water recovery system (and a new distillation unit) to the current hardware.

Par 4.4 Detail design and fabricate the approved preliminary design, and perform component and subassembly testing prior to installation in the integrated system.

Par 4.5 The compression distillation unit shall be capable of processing either urine and urinal flush water, or commode flush water and brine from a reverse osmosis unit renovating used wash water.

Par 4.6 Conduct two 30-day system tests; one 30-day test with the distillation unit processing urine and urinal flush water, and another 30-day test with the unit processing commode flush water and brine from a reverse osmosis unit renovating used wash water.

Par 4.7 Prepare and submit detail test plans for tasks 4.4 and 4.6, for the NASA MSC's approval, before these tasks are initiated.

While these objectives were being accomplished, CHEMTRIC was also preparing the preliminary design for a Space Station Prototype (SSP) water and waste management system, via a subcontract (on NAS9-10273) with the Hamilton Standard Division of the United Aircraft Corporation. This study indicated that additional experimental work should be performed to minimize the expendables required on a space station. Contract NAS9-9191 was, therefore, modified to include the following additional objectives.

*Paragraph in contract Exhibit "A" dated 1 December 1969

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Par. 3.2.1* Conduct a 30-day test of the urine loop to determine (1) the effect of solids concentration on water quality, and (2) the maximum concentration which permits stable operation.

Par. 3.2.2 Conduct a 30-day test of the urine loop to determine the minimum quantity of pretreatment chemical required.

Par. 3.3 Analyze, design and fabricate a vapor pyrolysis unit for the distillation unit - and conduct a 60-day test to determine the reduction in post-treatment expendables possible with vapor pyrolysis.

By agreement, the vapor pyrolysis unit was not fabricated and tested - because other improvements in the distillation system reduced the quantity of post-treatment expendables necessary to a relatively insignificant level. Instead, additional tests were performed with the distillation unit in the wash-water loop - to determine if the quantity of charcoal required to "polish" the permeate from the reverse osmosis (RO) module could be reduced by using a different germicide. When these tests indicated that an RO module is easily fouled by precipitates, contract NAS9-9191 was modified again to include the following objectives.

Par. 3.3.5.1 Conduct static tests to determine the occurrence and size distribution of suspended solids resulting from the use of various washing agents.

Conduct compatibility tests to determine the effect of washing agents on RO cell performance.

Conduct another wash-loop test to demonstrate design performance of a flush/wash water subsystem for a 30-day minimum operational period.

Par. 3.3.5.2 Agents to be used in these tests shall include, as a minimum, Miranol and Nutrogena soaps and Hyamine 1622 and Vancide BN germicides.

The processing rates originally specified for this program are as follows.

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*Paragraph in contract Exhibit "A" dated 25 November 1970.

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	lb/day	kg/day
Urine Distillation	30.6	13.9
Urine	13.8	6.3
Urinal Flush Water	8.0	3.6
Humidity Condensate	8.8	4.0
Fecal/Wash Distillation	13.2+	6.0+
Commode Flush Water	13.2	6.0
RO Cell Brine	TBD	TBD
Reverse Osmosis	316.8	144.0
Shower Water	158.4	72.0
Sink Water	105.6	48.0
Clothes Wash Water	52.8	24.0

Thus, the distillation unit was to be designed to process the urine, urinal flush water and humidity condensate anticipated with a 4-man crew - or the commode flush water anticipated with a 4-man crew, plus the brine from an RO cell renovating the wash water anticipated from 12 men. Later in the program some of these processing rates were modified to more accurately match the requirements anticipated on a modular space station.

1.3   Accomplishments

The work performed under contract NAS9-9191 yielded the following accomplishments - which assure the availability of an "effective" water and waste management system for manned space-craft. These accomplishments are listed in chronological order, according to subsystem, to justify the recommendations listed in Section 1.4; reference pages are in paranthesis.

1.3.1   Compression Distillation

A.   Developed a design which does not contain any external dynamic seals nor any external rotating parts. (p. 3-4)

B.   Demonstrated that the specific energy demand for this unit when processing treated water is less than 68 watt-hours/kg (31 watt-hrs/lb) - which is substantially less than demonstrated for any previously developed compression distillation unit. (p. 4-5)

C.   Demonstrated that 1.09% of the vapor generated must be used to purge the condenser of non-condensable gases which evolve from treated waste water. (p. 6-4)

D.   Demonstrated that water yields near 97% can be achieved with an externally-located solids dryer and "overboard" purging - but that the water distilled from the solids contains many contaminants which grossly affect the quality of the condensate. (pp. 5-7 & 5-13)

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E. Demonstrated that this unit can reliably process waste water which contains solids capable of passing through a 25-micron filter. (p. 5-9)

F. Demonstrated that this unit can be operated for 60 days at a four-man rate without any malfunctions or performance degradation - and if the motor-compressor coupling is improved, it's useful life will be greater than 180 days. (p. 6-4)

G. Demonstrated that the recycle-to-condensate flow ratio required to distill concentrated wash water is higher than for concentrated urine. (p. (6-4)

H. Demonstrated that if the unit is operated with untreated urine, the evaporator is easily fouled and the condensate purge rate must be increased to dispose of gases produced by putrifacation and/or fermentation. (pp. 7-8 & 7-9)

I. Demonstrated that if the evaporator is flushed with raw condensate prior to each shut-down, and if the proper recycle-to-condensate flow ratio is used, the unit will perform reliably with a recycle loop which contains up to 56% solids obtained from treated urine and urinal flush water. (p. 8-10)

J. Demonstrated that water yields near 97% can also be achieved when processing treated urine by simply accumulating recycle liquor in a replaceable tank. (p. 8-11)

K. Demonstrated that the overall heat transfer coefficient varies linearly from 120 cal/hr-cm^2-^{o}C to 30 cal/hr-cm^2-^{o}C as the solids content of the recycle loop is varied from 0 to 56%. (p. 8-11)

L. Demonstrated that the slip constant for the rotary-lobe compressor is 465 $\pm$ 35. (p. 8-14)

M. Demonstrated that a compressor with coated lobes can achieve the same performance as a compressor which utilizes a grease to minimize slippage. (p. 10-2)

1.3.2 Expendables

A. Demonstrated that if 2.25 grams of the developed pretreatment solution is added to the urine collected during each micturation - this urine will remain sterile for at least 10 days when stored at room temperature. (p. 7-5)

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B. Demonstrated that the 25μ filter, used to avoid plugging
    the distillation unit and it's recycle loop, has a useful
    life slightly more than 120 man-days when processing con-
    centrated urine and treated urinal flush water. However,
    under weightless conditions it's useful life may be less
    because "settling" of heavy fractions was observed in the
    units tested (p. 8-15)

C. Demonstrated that "as purchased" biological filters are
    plugged with solids after 188 man-days of use at outlets
    for flush and potable water. The same filters packed with
    a mixture of AgCl particles and glass beads have a useful
    life greater than 252 man-days when used to filter the raw
    condensate produced by the distillation of treated urine
    and urinal flush water. (p. 8-16)

D. Demonstrated that the useful life of an activated carbon
    filter located downstream of a biological filter contain-
    ing AgCl particles is at least 120 man-days, when proces-
    sing the raw condensate produced by the distillation of
    treated urine and urinal flush water. (p. 8-16)

E. Demonstrated that a deionizer can be presterilized, and
    that it can produce potable water for at least 120 man-
    days when fed carbon filtered and AgCl dosed condensate
    distilled from urine and treated urinal flush water.
    (p. 10-14)

F. Demonstrated that 61 milliliters of recycle liquor must be
    disposed of each man-day when the distillation unit is
    processing a mixture of urine and treated urinal flush
    water that has a solids content of 1.9%. (p. 8-17)

G. Demonstrated that septums used for withdrawing sterile
    water samples have a useful life of about 30 penetrations
    by an 18-gage needle.

H. Demonstrated that the useful life of a column packed with
    a mixture of AgCl particles and glass beads, for dosing
    carbon filtered condensate with AgCl ions, is greater than
    556 man-days (p. 10-14)

1.3.3 Reverse Osmosis

A. Demonstrated that Amicon's Diaflo UM-05 and Eastman's
    RO-97 membranes can be degraded by a common surfactant.
    (p. A-10)

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B. Demonstrated that cationic and anionic species in wash water can form sols and/or precipitates which can foul the surface of an RO membrane. (p. A-10)

C. Demonstrated that Vancide BN is more easily rejected by DuPont's B5 Permasep material than other germicides such as quartenary ammonium salts. (p. A-10)

D. Demonstrated that Hexachloraphene, a relatively insoluble germicide, easily fouls the surface of an RO membrane. (p. A-18)

E. Demonstrated that DuPont's B5 Permasep material is not degraded by the amphoteric surfactant Miranol C2M. (p. A-34)

F. Demonstrated that when a B5 Permasep unit is used to process 3μ -filtered wash water containing the recommended concentrations of Miranol C2M and Hyamine 1622 it has a useful life slightly more than 73 man-days, and the permeate can have more than 1000 ppm total solids and 800 ppm total carbon - because the fiber bundle in the Permasep unit tends to accumulate solids which increase the osmotic pressure and/or foul the fiber surfaces. (pp. 6-10, 6-14 & 6-18)

G. Demonstrated that when powdered Vancide BN is substituted for Hyamine 1622, and the dish water contains roast beef hash, the permeate obtained from a B5 Permasep unit has a total carbon content less than 300 ppm - but the unit is "fouled" in several man-days. (p. 9-9)

H. Demonstrated that liquid Vancide BN does not contain an insoluble residue like powdered Vancide BN - and therefore is the preferred germicide for use in wash water which is to be renovated by reverse osmosis. (p. C-4)

I. Demonstrated that Miranol C2M is more easily filtered and miscible with wash water ingredients than other amphoteric surfactants like Miranol JEM and Tegobetaine-C. (p. C-4)

J. Demonstrated that liquid Neutrogena Rainbath Body Soap Gel is more easily filtered and miscible than other personal skin cleaners like Neutrogena Bar Soap and Ivory Bar Soap. (p. C-22)

K. Demonstrated that the turbidity of used wash waters ranges from 130 to 50 Jackson Units - and that passage through a 100 millimicron filter is necessary to achieve a turbidity less than 1.0 Jackson Units. (p. C-24)

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L. Demonstrated that both the Westinghouse 4 X 4 RO module and DuPont B9 Permasep are compatible with a solution containing Neutrogena Rainbath Body Soap Gel, Miranol C2M-SF, liquid Vancide BN and AgCl ions - for at least 30 days. (p. C-33)

M. Demonstrated that the Westinghouse 4 X 4 module can produce permeate which has a COD in the range of 215 to 250 ppm - but the water yield must be in the range of 77% to process the used wash water anticipated from 6 men in one day. (pp. 10-7 & 10-15)

1.4 Recommendations

This report shows that the compression distillation unit developed under Contract NAS9-9191 is very reliable and efficient - and that the expendables required by this unit to produce sterile, potable water are relatively insignificant. However, this report also shows that additional testing is required to demonstrate that reverse osmosis is an acceptable process for renovating used wash water in an integrated water and waste management system. Thus, it is recommended that the NASA MSC continue to support the experimental evaluation of commercially-available RO modules, and consider supporting the development of an RO module specifically designed for renovating used wash water onboard manned spacecraft.

The two most promising RO modules evaluated during this program are the Westinghouse 4 x 4 and DuPont B9 units. The Westinghouse unit should be evaluated with a recycle loop which contains an 88μ cleanable filter, and provides the superficial velocity necessary to prevent surface fouling by agglomerated particulates; with this arrangement yields above 90% should be attainable, and the particulates collected in a filter which does not have to be changed as frequently as a 3μ prefilter. Surface fouling cannot be avoided in the DuPont B9 module; however, because of it's size, the module may have an acceptable useful life when operated without a fine prefilter - and thereby, have a lower effective weight penalty than the Westinghouse module which requires recycle power and a replaceable particulate filter. Both modules should be extensively evaluated, unless it is decided that Vancide BN is not acceptable - in which case only the Westinghouse module, which is compatible with Hyamine 1622, should be tested.

Additional work recommended to further improve the compression distillation unit and it's supporting components are:

A. Develop a peristaltic pump which contains gear-driven rollers to avoid abrasive wear and collapsing of the tubing.

B. Incorporate a magnetic coupling in the distillation unit so that the drive motor can be located externally - to facilitate replacement, and avoid the need for an internal coupling.

C. Redesign the demister so that it rotates within the evaporator bowl, to reduce the carryover which occurs when the distillation unit processes concentrated wash water containing foaming surfactants and/or germicides.

Finally, the compression distillation unit design has been developed to the level where the development of a flight-qualified model can be confidently undertaken. Also, it's performance under weightless conditions should be even better than demonstrated in the laboratory - because gravity affects the heat transfer coefficients, and tends to promote carryover. Consequently, it is also recommended that the NASA consider developing a compression distillation experiment for the Sky Lab and/or Space Shuttle programs - so that the performance of an automated unit under weightless conditions will be available prior to the development of an operational unit for a modular space station.

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PRELIMINARY TESTING & ANALYSIS

Work on this program was started while the prototype hardware developed by Marquardt, under contract NAS9-5119, was being refurbished and tested under contract NAS9-9014. Therefore, the first task undertaken was an experimental evaluation of reverse osmosis membranes for renovating used wash water. When it was discovered under contract NAS9-9014 that the compression distillation units developed by Marquardt could not dry solids, contract NAS9-9191 was modified to include a parallel investigation of capillary media for use in an externally-located solids dryer. Next, the contract was modified to include the development of a new compression distillation unit. The results of these preliminary experimental investigations, and the analyses performed to preliminary design a new compression distillation unit, are summarized in the following subsections.

2.1   Reverse Osmosis Tests

A detailed summary of the preliminary experimental work performed on reverse osmosis (RO) membranes, surfactants and germicides is presented in Appendix A. From the results obtained it was concluded that (1) Miranol, an amphoteric surfactant, should be used to avoid the formation of sols and/or precipitates when using RO to renovate used wash water, (2) DuPont's Model B5 Permasep is the best RO material available for renovating used wash water, (3) recycling brine to decrease osmotic pressure does not provide any significant advantage over single pass operation, and (4) Vancide BN is the preferred germicide to use in clothes and dish water which is to be renovated by RO.

Additional recommendations, formulated from the experience gained by performing these experiments, are described as follows.

1.   Used wash water must be filtered before it is fed into an RO cell, to avoid fouling the membrane surface with gross quantities of suspended solids. DuPont recommends a 5 micron prefilter for their B5 Permasep material. Since these types of filters have relatively low capacities for solids, a coarser filter should be provided upstream of the 5 micron filter to minimize the total weight of expendables. If the coarse filter can be cleaned in-place, even greater weight savings will be realized.

2.   RO cells must be operated at relatively high pressures to produce high quality permeate. When used in spacecraft (i.e., relatively low processing rates) a positive displacement type of feed pump is required to minimize electric power demand.

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3. Reverse osmosis units should not be required to recover more than about 95% of the used wash water; otherwise, they may be prematurely fouled by solids precipitated as the result of excessive brine concentration.

4. The water permeability of RO membranes is a strong function of temperature (e.g., 3% increase for every 1°C increase in temperature); therefore, with a constant feed rate the brine should be bled-off at a constant rate to avoid the excessive brine concentrations which would result when the operating temperature is higher than nominal.

5. Used wash water contains substantial quantities of microorganisms and nutrients. This water should be pasteurized and/or dosed with a germicide before it is processed in an RO cell. In either case, the RO cell permeate should be dosed with a residual-acting germicide so that renovated wash water is sterile when drawn for use.

All of these recommendations were incorporated in the upgraded IWWM hardware, as described in Section 3.3 of this report.

2.2 Compression Distillation Analysis

2.2.1 Previous Designs

The most successful compression distillation unit tested before this program was the unit developed by Nuccio and Jasionowski under contract AF33(615)-2124*. This unit was continuously operated for 17 days (14 days with treated urine) to demonstrate that (1) recycling the feed liquor maintains the evaporator surfaces in a clean condition, (2) more than 85% of the input water can be recovered with a specific energy demand less than 56 watt-hours per pounds, and (3) the recovered water conforms to the 1961 Drinking Water Standards of the U.S. Public Health Service. This unit produced at least 1.25 pounds of condensate per hour using (1) approximately 3.0 ft^2 of heat transfer area (common condenser and evaporator wall), (2) a rotary-lobe compressor (0.0105 ft^3/rev) driven at 3160 rpm, and (3) a 208 V/400 Hz/3 motor rated at 60 watts to drive the compressor, rotate the evaporator and condenser bowls at 60 rpm, and operate the internally-located liquid pumps. Thus, this unit is capable of processing the urine, urinal flush water and humidity condensate anticipated from a 4-man crew (30.6 #/day), or the commode

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*Nuccio, P. P. and Jasionowski, W. J., Automatic Water Recovery System, AMRL-TR-67-155, March 1968.

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flush water and ... specified for contract NAS9-9191 (13.2 + .05 x 316.8, or ...). However, it was decided that a new compression dis... unit should be developed under this program, to avoid the ... out problems encountered with the Air Force unit.

The mechanical problems encountered with the Air Force compression distillation unit are described as follows, along with the improvements recommended for a new unit.

1. The internally-located feed, recycle and condensate pumps use cam-operated pistons provided with O-ring and/or "slipper" seals; abrasive solids in the recycle liquor cause the recycle pump seals to have a useful life slightly more than 17 days. To avoid this problem, the pumps should be (1) designed to operate with abrasive solids, and (2) externally located so that the distillation unit does not have to be opened to replace a pump if it does fail.

2. The speed reducers located on each end of the electric motor, to drive the compressor and rotate the bowls, have a useful life of about 14-days. An improved unit should drive the compressor directly from the motor - and use a chain-and-sprocket or belt-and-pulley arrangement for rotating the bowls.

3. The custom-designed compressor uses relatively small-diameter cast-aluminum lobes mounted on relatively large steel shafts; thus, the lobes axis of symmetry are easily skewed by creep and/or any set-up loads - so that the lobes interfere with each other. A larger diameter compressor with cast-steel lobes should be used to avoid this problem.

4. The two O-ring type of dynamic seals required in the Air Force unit experience excessive wear due to poor concentricity of the sealing surfaces. An improved unit should avoid all dynamic seals, or at least use carbon-type face seals as done in the Marquardt unit.

Additional improvements recommended as the result of Marquardt's work under contract NAS9-1680 are:

1. The cylindrical evaporator bowl should be provided with a ring-type dam at the drain end - to assure wetting of the entire evaporation surface, and control the film thickness. If this is not provided, the feed liquor will flow through the "valleys" created by the eccentricities - which are unavoidable when fabricating a thin-wall cylinder by rolling and welding.

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2. The rotational speed of the evaporator bowl must be sufficient to produce a centrifugal force greater than 1-g at the inner surface; otherwise, the feed liquor will "fall-off" the upper part of the evaporator surface when subjected to earth's gravity.

All of these improvements were incorporated in the new compression distillation unit which is described in Section 3.2.

2.2.2 Performance Goals

The design requirements specified in contract NAS9-9191 state that the compression distillation unit must be compatible with a 4-man urine loop, and a 4-man fecal loop which is fed the brine from a 12-man wash water loop. With an RO cell operating to achieve a water yield of 95%, the daily quantity of waste water to be processed in the fecal loop is only 13.2 + 0.05 x 316.8, or 29.0 #. In the urine loop, the distillation unit must process 30.6 #/day; therefore, the new distillation unit was designed primarily to achieve the performance required when it is installed in a 4-man urine loop.

To assure compliance with the requirements anticipated for a modular space station, it was decided that the following performance goals should be established in addition to those specified by the contract.

Minimum Yield: 98% of the feed water.

Recovery Rate: 30.6 x 0.98, or 30.0 #/day when operating with an ambient temperature of $60^{\circ}F$.

Specific Energy: Less than 55 watt-hours/# when operating with an ambient temperature of $72^{\circ}F$.

Maintenance: No more than once every 10 days.

2.2.3 Heat Transfer Area

The heat transfer area required in a compression distillation unit is usually computed from the following equation.

$$A = \dot{m} \times h_{fg}/U \times \Delta t, \; ft^2$$

$\dot{m}$ = distillation rate, #/hr

h_{fg} = latent heat of vaporization, Btu/lb

U = heat transfer coefficient, $Btu/hr\text{-}ft^2\text{-}^{\circ}F$

Δt = film potential, $^{\circ}F$

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The problem is to select the optimum film potential (Δt), considering the characteristics of the vapor compressor, and then estimate the actual heat transfer coefficient (U) which will be achieved. In addition, the effectiveness of the designed area (surface) must be considered - because eccentricity, surface irregularities, vapor superheat and variation in boiling point of the liquor all affect local heat transfer.

Berninger, Charanian and Bambenek[*] presented the results of a trade-off study which shows that the optimum film potential is $6^{\circ}F$. This result is not surprising because a Δt of $6^{\circ}F$ or more is required to obtain nucleate boiling, and compressor power penalty is higher than the weight saving made possible if a larger Δt is used. Thus, for the new distillation unit it was decided to use a film potential of $6^{\circ}F$.

The thermal resistances of the condensation film and the metal wall separating the condenser and evaporator in a compression distillation unit can be ignored - because they are one to two orders of magnitude less than the resistance of the vaporization film. Classical data on water boiling heat transfer indicates that film coefficients above 150 Btu/hr-ft^2-$^{\circ}F$ should be realized with a Δt of $6^{\circ}F$, and gravity acting to displace bubbles generated on the heated surface. Using the Nusselt expression for film evaporation, Marquardt[**] concluded that with a Δt of $6^{\circ}F$ and a centrifugal force of 5 - 6 g's - the film coefficient should be near 150 Btu/hr-ft^2-$^{\circ}F$. However, the compression distillation units developed up to this time for processing urine never experienced film coefficients near 150 Btu/hr-ft^2-$^{\circ}F$; instead, the units developed by Beyerman et al. and Nuccio and Jasionowski both demonstrated that the average coefficient is near 75 Btu/hr-ft^2-$^{\circ}F$ - or that the effective heat transfer area is about one-half of the actual area.

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[*] Berninger, J. F., Charanian, T. R. and Bambenek, R. A. Water Reclamation via Compression Distillation, Paper presented at the ASME Aviation & Space Division Conference, Los Angeles, March 1965.

[**] Beyerman, E. E., Ellis, G. E. and Merzenick, J. B. Waste Management Program, Final Report on Contract NAS9-1680, Marquardt. Van Nuys, February 1965.

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An effective [illegible] to only one-half the actual area is understandable with [illegible] and Jasionowski's unit because (1) the cylindrical e[illegible] did not contain a ring-type dam at the drain-end to assure wetting of the entire surface, and (2) the rotational speed was not sufficient to maintain a liquid film on the upper surface when operating in a one-g environment. However, Beyerman's unit was designed to avoid these problems. The only plausable explanations for this discrepancy are: (1) Beyerman's test durations were too short to achieve thermal equilibrium, (2) Beyerman's dam was too low to compensate for eccentricity, and/or (3) the viscosity of concentrated urine is too high to achieve nucleate boiling coefficients. Because of this situation it was decided to design the new distillation unit so that it would achieve the desired recovery rate even if the resultant film coefficient was 75 Btu/hr-ft^2-^{o}F. Consequently, the required heat transfer area was calculated as follows.

$$A = 1.25 \times 1065/75 \times 6 = \underline{2.98 \text{ ft}^2}$$

The heat of vaporization was taken for water at 51°F, which is the minimum evaporator temperature anticipated when the feed liquor is 30% solids and the ambient temperature is 60°F. Condenser temperature tends to stay a few degrees above ambient temperature; therefore, to assure attainment of the minimum recovery rate it is best to assume that condenser temperature will equal ambient temperature.

2.2.4   Compressor Lobe Length

At the beginning of this program it was planned to have the Sutorbilt Corporation custom-design and fabricate a rotary-lobe compressor, as they did for the unit developed under contract AF33(615)-2124. However, it was decided to purchase and modify an off-the-shelf compressor when it was learned that Sutorbilt could not deliver a custom-designed unit until six months after the design requirements were established. The commercially available unit selected was the Gardner-Denver Co. (Quincy, Illinois) Model 2PDR4 - which has a gear diameter of 2-1/2 inches and a lobe length of 4 inches. The analytical problem to be solved was calculating the required reduction in lobe length to minimize power, and still achieve the desired recovery rate.

Rotary-lobe compressors are rated in terms of displacement per revolution; the model selected displaces 0.033 ft^3 per revolution. However, actual performance is a function of gas or vapor properties, pressure differential and rotational speed. Since back flow through the end and tip clearances is a weak function of rotational speed, the current industrial practice is to compute these losses in terms of a "slip" rpm, and then drive

the compressor at the rpm required to achieve the desired "net" flow. For the new distillation unit it was decided to direct-drive the compressor with a two-pole, 60 cycle motor - to (1) avoid gearing, (2) achieve reasonably high volumetric efficiencies, and (3) permit the use of readily available motors and power supplies. Thus, instead of computing the rpm required, the problem was to compute the lobe length necessary to achieve the desired net flow with a 3200 rpm[*] motor.

The equation relating slip rpm to vapor properties and pressure differential is:

$$S = K \sqrt{k/3.5(k-1)} \ \sqrt{0.075 \upsilon} \ \sqrt{\Delta p}$$

Where K = the slip constant
      k = ratio of specific heat
     υ = inlet specific volume, ft^3/lb.
  Δp = pressure differential, psi

Charanian et al.[**] correlated available slip data on Sutorbilt rotary-lobe compressors and developed the following expression for slip constant (K).

$$K = 1000(10)^{-GD/4}$$

Where GD = gear diameter, inches

Since this expression was developed from published data on a wide range of industrial compressors, a K value computed from this equation must be considered as being a "rough" estimate of the actual value to be obtained with a new compressor. Thus, for the new compressor the maximum slip constant was estimated to be:

$$K = 1000/10^{2.5/4} = \underline{240}$$

then, the maximum slip rpm was estimated as being:

$$S = 240 \ \sqrt{1.32/3.5(1.32-1)} \ \sqrt{0.075 \times 1644} \ \sqrt{0.257 - 0.184}$$

$$= 240 \times 1.09 \times 11.1 \times 0.27 = \underline{785} \ rpm$$

---

* Typical rated speed of small 2-pole, 60 cycle motors which develop reasonably good starting torques.

** Charanian, T. R. et al., Extended Mission Apollo Study on Water Reclamation, Waste Management & Personal Hygiene, GARD Report 1276-6070, GARD: Niles, November 1965.

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For this calculation it was assumed that the condenser and evaporator are saturated at 60°F and 51°F, respectively - to provide a Δt of 6°F for nucleate boiling and 3°F for the boiling point elevation (depression)* anticipated when the evaporator liquor contains 30% dissolved solids.

The minimum "productive" rpm of the compressor was then predicted to be 3200 - 755, or 2415 rpm - which corresponds to a volumetric efficiency of 2415/3200, or 75.5% (a reasonable value for subatmospheric applications).

Finally, the required maximum length of the lobes was calculated as follows - knowing that the displacement with 4-inch long lobes is 0.033 ft^3/rev.

$$L = \dot{w}\upsilon/60N \times 0.033/4$$
$$= 1.25 \times 1644/60 \times 2415 \times 0.033/4$$
$$= \underline{1.72 \text{ inches}}$$

2.2.5 Centrifuge Speed

The cylindrical evaporator and condenser must be rotated to (1) retain the liquor on the evaporator surface, and (2) force condensate off of the condenser surface - when operating in a weightless or a non-weightless state. The highest rotational speed is required when operating on the ground because the radial acceleration of the liquor must exceed the acceleration of gravity - to keep the liquor against the rotating evaporator surface. The axis of rotation, of course, must be normal to the gravitational vector to maintain a fairly uniform film of liquor on the evaporator surface.

To avoid the pump-out problems encountered with the AMRL unit it was decided to utilize externally-located peristaltic pumps, and stationary pick-up tubes within the evaporator and condenser. Thus, the rotational velocity of the condensate and the concentrated feed liquor could be used to force these fluids to their respective pumps, which would then produce the pressure necessary to force these fluids to accumulators. The required rotational speed was then calculated as follows.

The head required at the impact tube inlet was set at 7 inches, because that is the maximum condenser centrifuge radius. The minimum velocity required to generate that head is given by the expression:

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*Putnam, D. F., Chemical and Physical Properties of Human Urine Concentrates, NASA CR-66612, April 1968.

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$$V_{min} = \sqrt{2g \times hd} \times 60$$

$$V_{min} = \sqrt{2 \times 32.2 \times \tfrac{7}{12}} \times 60$$

$$V_{min} = 367 \text{ ft/min}$$

and the required minimum speed of the liquid is:

$$V_{min} = \frac{V_{min}}{\text{circumference}} = \frac{\text{ft/min}}{\text{ft/rev}}$$

$$V_{min} = \frac{367 \times 12}{2 \times 7 \times \pi} = 100 \text{ rpm}$$

The minimum radial acceleration necessary is equivalent to one-gravity, or 32.2 ft/sec^2, and is related to radius and speed by the expression:

$$A_{min} = r \omega^2 \text{ ft/sec}^2$$

Transposing, the required minimum speed is:

$$\omega_{min} = \sqrt{\tfrac{g}{r}} \text{ rad/sec}$$

$$\omega_{min} = \sqrt{\tfrac{32.2}{7/12}}$$

$$\omega_{min} = 7.4 \text{ rad/sec or 71 rpm}$$

The minimum speed required to generate the necessary velocity pressure at the impact tube is greater (100 rpm), and therefore, governs the design.

Some relative motion will prevail between the liquid and the centrifuge, requiring that the centrifuge be driven at a speed greater than the calculated liquid speed. Based on previous experience with probe-type urine-gas separators, it was concluded that the design rpm should be at least twice the theoretical rpm. Design speed of the centrifuge, therefore, is 205 rpm.

2.2.6 Motor Power

Motor selection should be based upon known power requirements of previous compression distillation units. In this case, empirical data is more dependable than arithmetically summing the many small power sink calculations, each of which must originate with at least one assumption.

The AMRL Unit, which displayed the best performance to date, requires 55 watt-hours per pound of water recovered from liquor concentrated to thirty-percent solids. The new unit designed

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without dynamic seal friction will require less energy per pound of water recovered from concentrated waste. An estimated 52 watt-hours per pound multiplied by the minimum recovery rate of 1.25 lb per hour establishes the minimum motor size, 65 watts. It is recognized, however, that the recovery rate will be higher than the minimum under virtually all operating conditions, because all sizing calculations are based on the most pessimistic set of circumstances which might prevail. Actual motor power required would exceed the minimum to accommodate the higher water recovery rates. Two motor selections were made, therefore, to span the power range anticipated. A standard induction motor (Globe Model #79A159) rated at 85 watts was selected as the high-power machine, and a modified version of the same motor derated to 65 watts chosen for the low-power requirement. In operation, power input to an induction motor is nearly linear with load within 20 to 30% on either side of rated load. With these two selected motors, therefore, any power requirement between 50 watts and 100 watts could be delivered without a penalty in motor efficiency.

2.2.7 Summary

The analyses performed prior to the detail design of a new compression distillation unit concluded that this unit should be an improved version of the unit previously developed by Nuccio and Jasionowski under Contract AF33(615)-2124. In addition, it was concluded that this unit should be capable of recovering 1.25 lb of water per hour when the ambient temperature is only 60°F, and the concentration of dissolved solids in the recycle loop is 30%. To assure this performance, the design calculations indicated that the unit must have the following values for the critical design parameters.

Heat Transfer Area:	2.98 ft^2
Compressor Lobe Length:*	1.72 inches
Centrifuge (Bowl) Speed:	>200 rpm
Motor Power:	65 to 85 watts

As shown later in this report, the new distillation unit does achieve the design performance; however, substantially higher performance was achieved by operating the unit in a closed cabinnet so that the condenser temperature is approximately 20°F higher than the ambient temperature.

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*Gardner-Denver Model 2PDR4

C H E ₀M T R I C

2.3 Solids Drying Tests

A detailed summary of the preliminary experimental work per-
formed on drying urine solids is presented in Appendix B. From
the results obtained it was concluded that:

1. The drying temperature should be limited to 160°F, to
   avoid gross thermal decomposition of urine solids such
   as urea.

2. When vacuum dried at 160°F, the urine liquor is concen-
   trated to 72% solids.

3. The most effective capillary media tested is Nylonge
   (a nylon reinforced cellulose sponge), which has a
   measured free volume of 64%.

4. The "active" drying zone is limited to a distance of
   1 inch or less from the heater surface.

5. The terminal capacity of a solids dryer equals the
   total void volume within the active drying zone.

The total quantity of solids anticipated during distilla-
tion tests was:

Urine   = 0.52 # solids/day
Urine Pretreat: = 0.13
   Total for Urine Loop   = 0.65 # solids/day

Commode Flush Pretreat: = 0.13 # solids/day
Wash Water   = 0.42
   Total for Fecal/Wash Loop 0.55 # solids/day

Therefore, the solids dryer was sized for the urine loop, which
should have a higher solids input than the fecal/wash loop.

The void volume required in the solids dryer, to support a
30-day test of the urine loop, was estimated as follows.

$$V = \frac{0.65 \ \#S/D \ \times \ 30 \ D}{0.72 \ \#S/\#1 \ \times \ 1.35^* \ \times \ 62.4 \ \#1/ft^3} = 0.322 \ ft^3$$

Since the void volume of Nylonge is 64%, the total sponge volume
required for each 30-day test was estimated to be 0.5 ft3.

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*Estimated specific gravity of liquor containing 72% solids per
  Putnam's data (NASA CR-66612).

C H E M T R I C

SYSTEM DEFINITION

The hardware available at the beginning of this program was the Integrated Waste Management System (IWMS) developed by Marquardt under Contract NAS9-5119, and upgraded and tested by AMGLO under Contract NAS9-9014. Figure 1 shows that this system consisted of two loops - i.e., a urine loop and a fecal loop. Each loop contained (1) a waste collection subsystem, (2) a compression distillation subsystem, (3) a waste and water holding tank, and (4) a post-treatment subsystem. This system was upgraded under Contract NAS9-9191 by (1) adding a wash water recovery subsystem to the fecal loop, and (2) fabricating a new distillation subsystem for use in either loop. Thus, with only one distillation subsystem available, only one loop could be tested at a time.

Additional changes made to the IWMS are:

1. The macerating-type of fecal collector (Hydro-John) was replaced with a two-stage collector furnished by the Hamilton Standard Division of the United Aircraft Corporation. This collector separates feces from the anal wash, and therefore, avoids the plugging problem encountered on Contract NAS9-9014.

2. The urine collection subsystem was discarded because the paddle-wheel type of liquid-gas separator was unreliable - and it was decided unnecessary to demonstrate the applicability of previously developed rotating-bowl separators. A graduated cylinder and peristaltic pump were used to feed waste water into the waste holding tank.

3. The system was replumbed so that the supporting structure contained only one loop - which could be operated as a urine loop or a fecal/wash loop.

Figure 2 shows two views of the upgraded hardware, as it existed at the end of this program. The two-stage fecal collector, shower stall and washing machine used for fecal/wash loop tests are not shown in these views.

The arrangement of the hardware was modified between test series as necessary to simulate a urine or fecal/wash loop, and to accomplish the specific objectives of each test series. The arrangements tested are described in the sections of this report which summarize each test series. The major components used in these tests are described in the following sub-sections.

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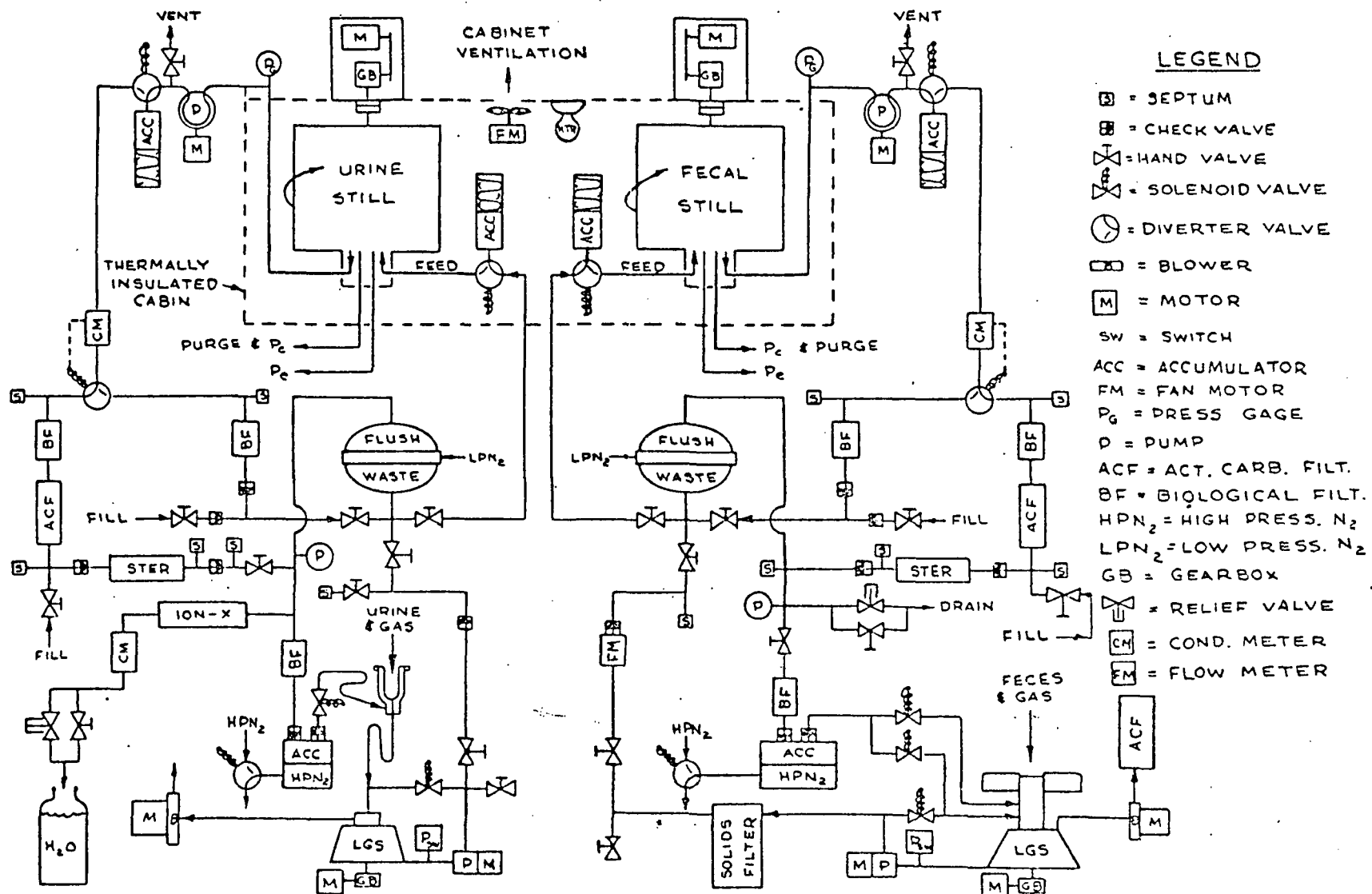
Figure 1   SCHEMATIC OF ORIGINAL TWO-LOOP SYSTEM
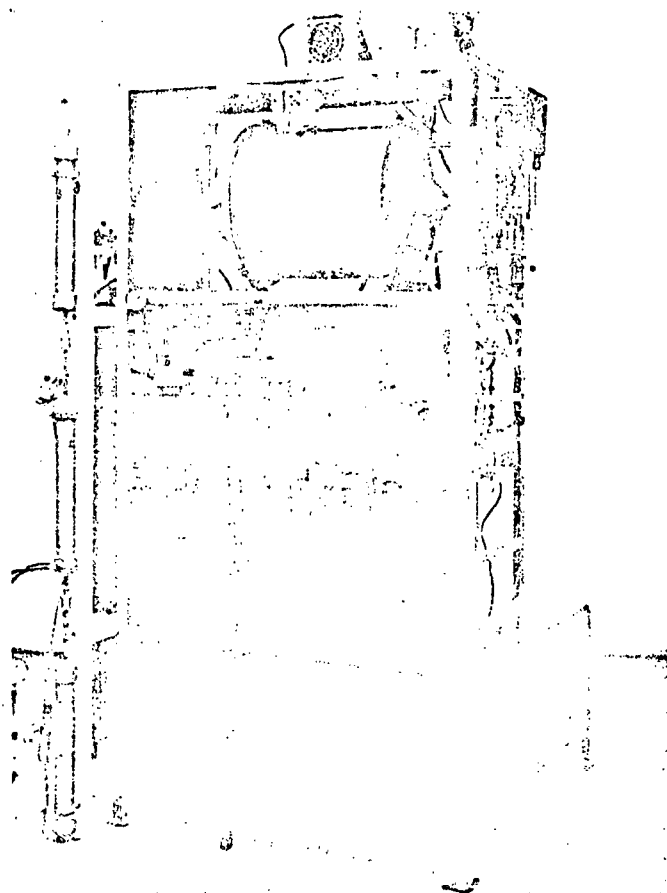
(DISTILLATION SIDES)   (REVERSE OSMOSIS SIDES)
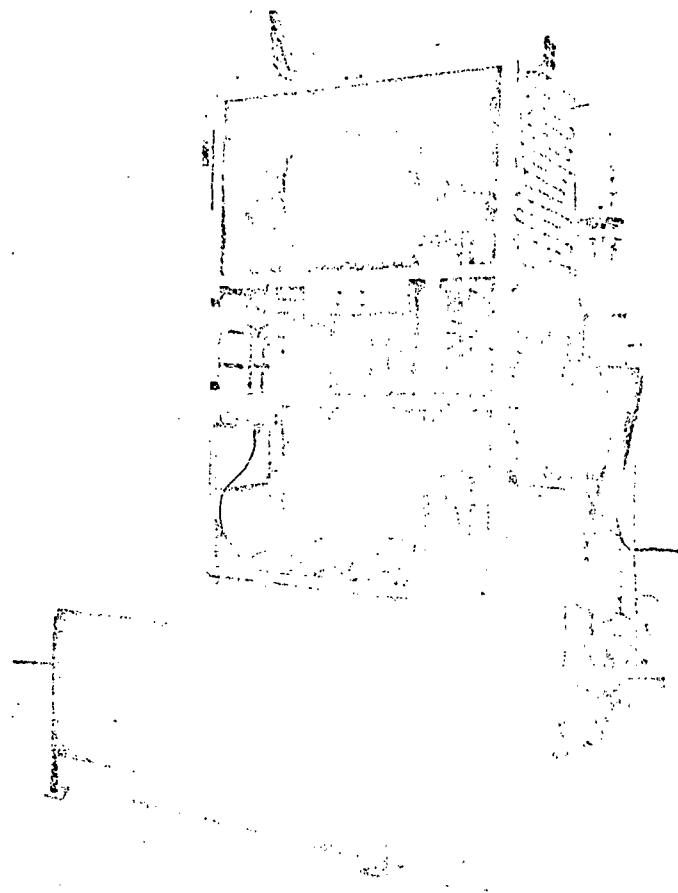
Figure 2   VIEWS OF THE UPGRADED HARDWARE

3.1 Distillation

The primary design goal for this unit was to avoid all of the mechanical problems encountered with previously developed compression distillation units - and demonstrate that compression distillation can be a reliable, as well as the most efficient process for recovering water onboard manned spacecraft. To achieve this goal a macroscopic analysis of the required mechanical functions was made first - before individual component parts were designed to perform those functions. It was recognized that only the compressor rotors and the centrifuge were necessary dynamic parts; all other functions could be performed by stationary members. This represents a major departure from all previously-designed vapor compression units because this new unit could be designed primarily as a stationary structure rather than as a rotating device.

The most significant design feature of the new distillation unit is that connections for transfer of liquids, instrumentation signals and electrical power can be made directly through stationary members. Dynamic seals and slip rings, which are always potential problem areas, are eliminated in the new design. By working within this fundamental concept an inherently reliable vapor compression distillation unit was designed. Figure 3 shows that this unit has the following unique design features.

1. The electric motor is located within the stationary shell (see Figure 4). The motor drives the compressor rotors directly, without the need for dynamic seals or speed reducers.

2. The compressor housing is stationary and is part of the central shaft (see Figure 5), which supports the rotating evaporator and condenser shell (which comprise the centrifuge).

3. Fluid connections are made to the unit through the stationary shaft at the end opposite the compressor. All passage surfaces are stationary including the waste water feed line - which terminates as a large diameter ring at the motor end of the evaporator. Waste flows from holes in the stationary ring onto the rotating evaporator surface.

4. The evaporator bowl contains a ring-type dam at the end opposite the feed ring - to assure wetting of the entire evaporator surface (see Figure 6). The height of the dam is greater than the maximum eccentricity of the cylindrical evaporator.

Figure 3   DISTILLATION UNIT ASSEMBLY DRAWING
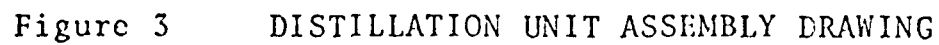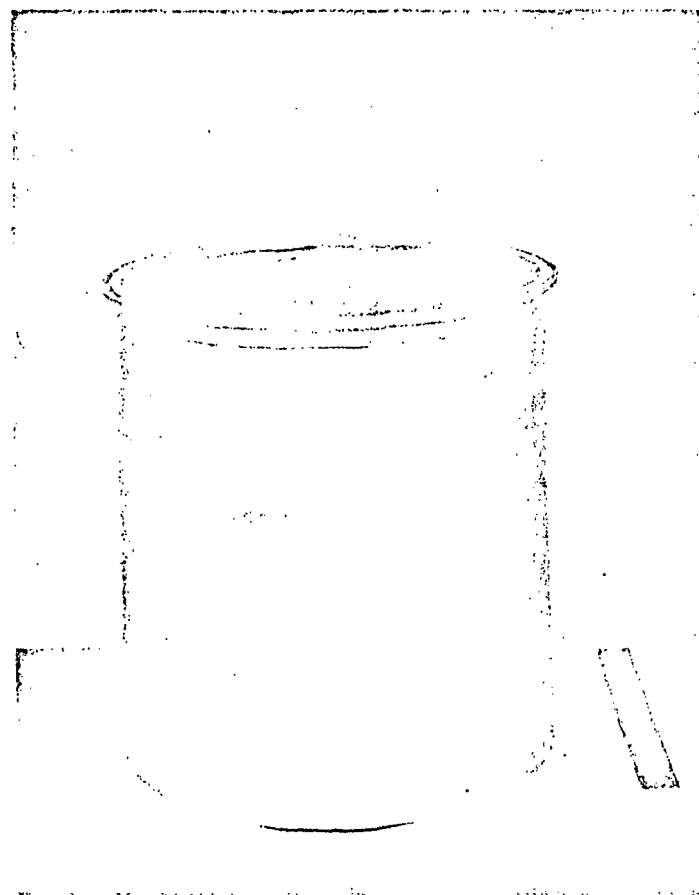
Figure 4   STATIONARY OUTER SHELL   Figure 5   COMPRESSOR & CENTRAL SHAFT
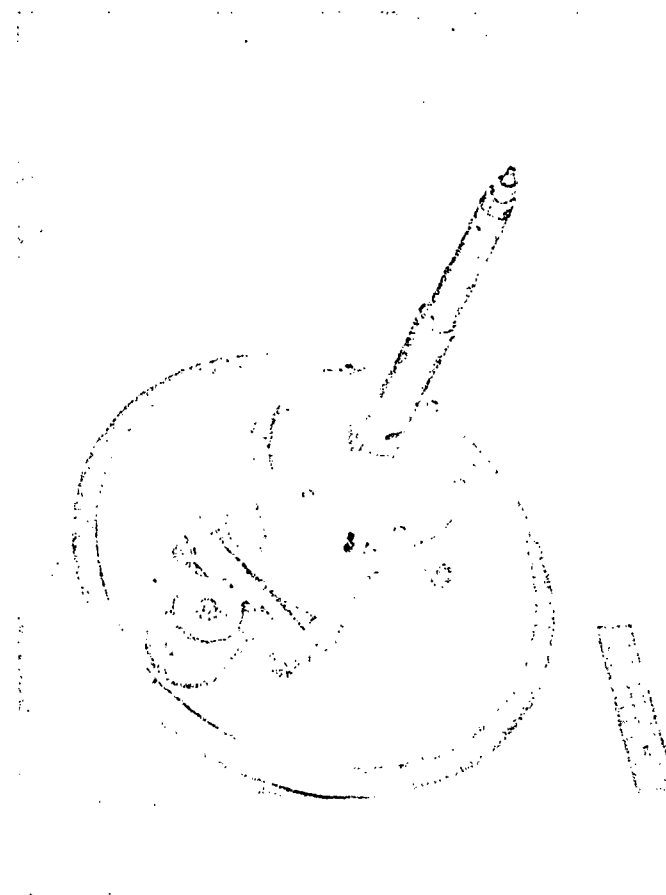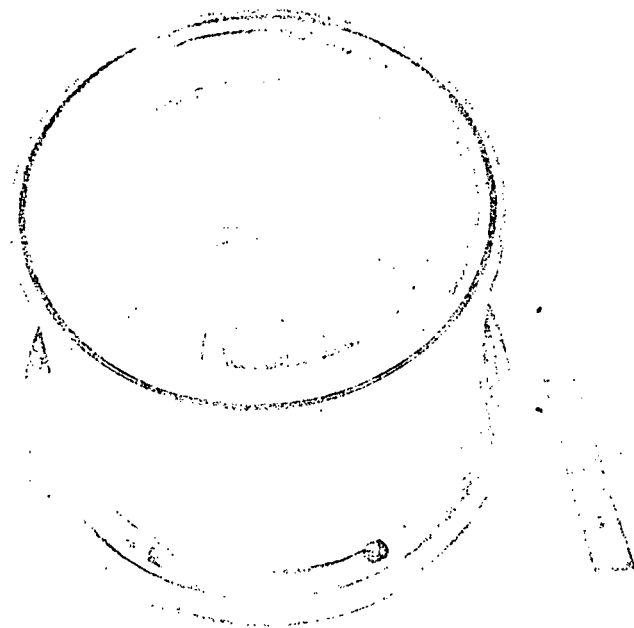
Figure 6   EVAPORATOR BOWL   Figure 7   CONDENSER SHELL
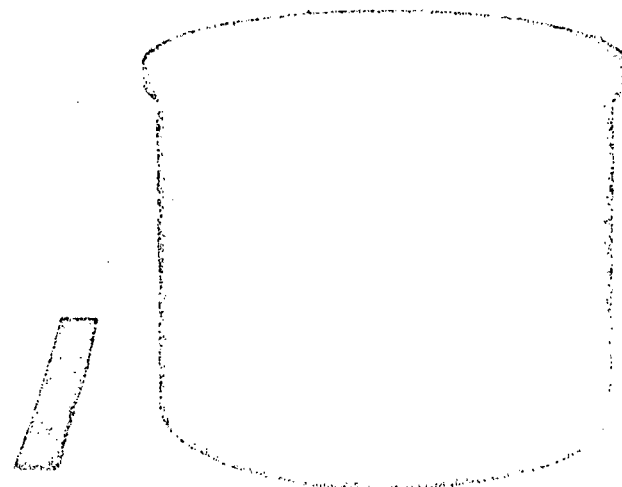
5. Excess feed flows through holes in the dam into the annular sump, where it is picked-up by a stationary impact tube and transferred through the stationary shaft to an externally located recycle pump. Condensate is removed by a similar tube originating in the condenser annular sump.

6. The condenser (see Figure 7) is purged through the stationary shaft, from the end opposite the vapor inlet - to avoid dead spaces and aid in the transport of vapor.

7. The desired liquid level in the evaporator is maintained automatically because the feed input rate is less than the removal capacity of the recycle pick-up and pump combination. However, a high-level sensor is provided in the evaporator to signal an upset condition.

8. The condenser is not pneumatically isolated from the outer shell; a small amount of vapor must leak to the outer shell where it condenses (to close the thermodynamic cycle), and is delivered to the condenser as a liquid via a third pick-up tube fixed to the rotating centrifuge. (The tube is visible in Figure 7.)

9. The centrifuge is driven by a non-lubricated cogged belt and O-ring speed reducer (shown in Figure 8). The O-ring drive is unconventional but ideally suited to the requirements in this application for two important reasons: (1) it can readily compensate for eccentricity and axial misalignment and (2) it can damp torsional vibrations originating at either the high-speed or low-speed member. The power transmitted through the O-ring is less than 5 watts mechanical, and no failure or measurable wear has occurred in approximately 2000 hours of operation with the original O-ring.

3.2 Solids Dryer

Section 2.3 concludes that the solids dryer (or concentrators) must contain 0.5 ft^3 of Nylonge media to dispose of the maximum quantity of solids anticipated during any 30-day test. Since two 30-day tests were originally specified, a total of 1.0 ft^3 of media was required to avoid "cleaning" a dryer between test series. Another self-imposed requirement was to design the dryer so that tests could be performed with gravity assisting or preventing the "breakthrough" of liquor. To accomplish these objectives a dryer was designed to contain two 0.5 ft^3 compartments, with an electrically heated plate separating the halves.

C H E M T R I C

O-RING
DRIVE BELT

COMPRESSOR
TIMING
GEARS

COMPRESSOR
LOBE SHAFTS

MOTOR

3200
RPM

3200
RPM

200
RPM

COGGED
BELT

CENTRIFUGE

DISTILLATION
UNIT - STATIONARY
VACUUM VESSEL

900
RPM
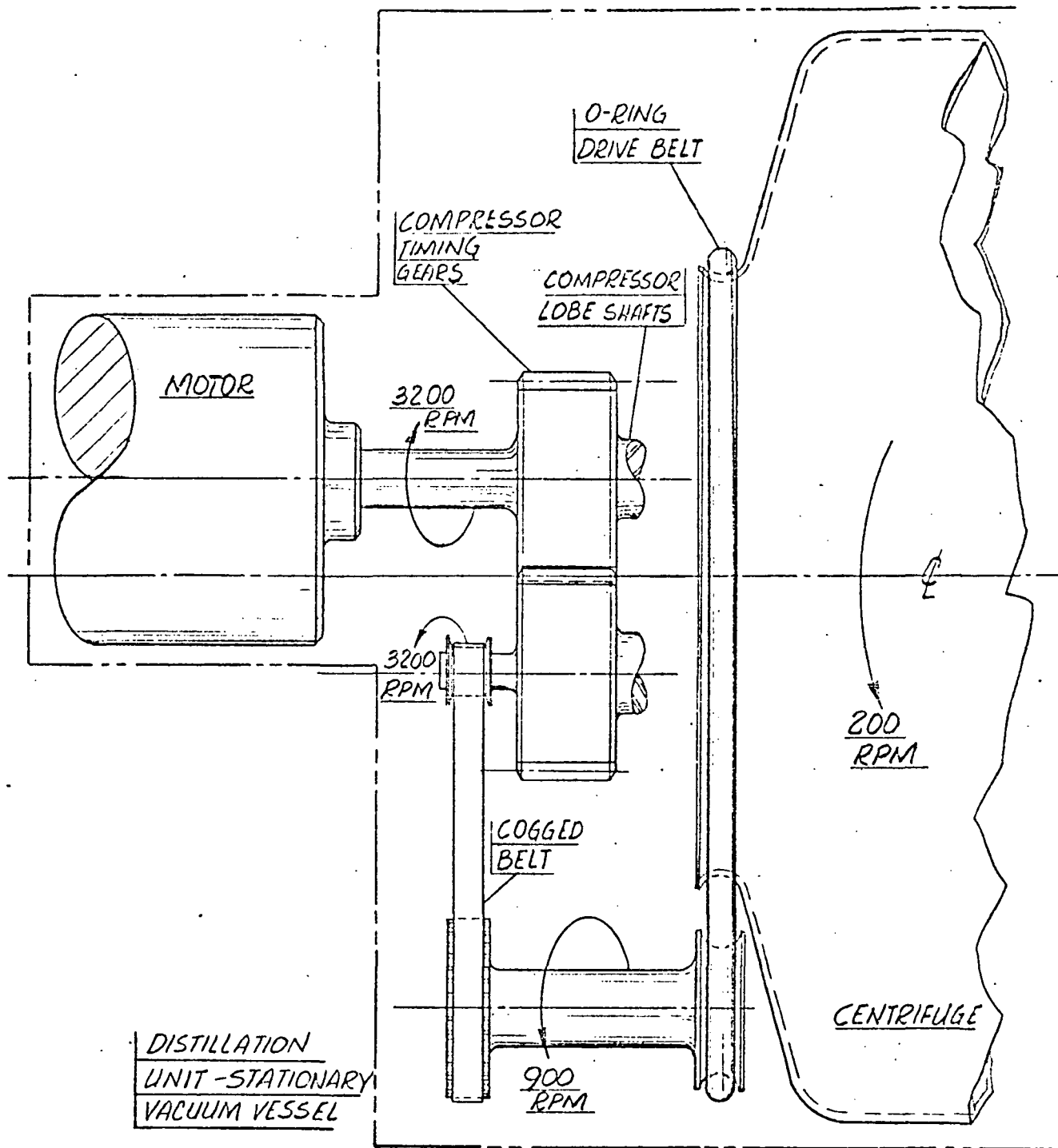
Figure 8   COMPRESSOR & CENTRIFUGE DRIVE

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Figure 9 shows the solids dryer partially disassembled after test. The cylinder and end caps are stainless steel spinings - fastened together by screws, and sealed with O-rings.

The heat required to concentrate liquor from 30% to about 72% is provided by five electric resistance heaters wired in parallel and sandwiched between the two stainless steel plates, which form one end of each compartment. The heaters are electrically insulated with silicon-rubber. Glass bead thermistors imbedded in the insulation are provided to control the heater temperature near 160°F, through a solid-state proportional controller.

As shown in Figure 9, each compartment contains longitudinal baffles (type 304 S.S.) which form slots for the capillary media - and serve as extended heat transfer surfaces so that the maximum distance to any point in a sponge is no more than 1 inch. The baffles are perforated to facilitate the transfer of liquor between compartments, and provide a multiplicity of low resistance paths for vapor to escape. Concentric tubes are located in each head so that both the point of liquor entry and vapor escape are on the axis of symmetry.

Thermocouples penetrate the cylinder to sense the temperature at three radial and three longitudinal locations.

3.3 RO Subsystem

Commercially available components were purchased to assemble this subsystem, as illustrated in Figure 10. The major components were selected as follows.

3.3.1 RO Processor

DuPont's B-5 Permasep material reportedly* has a surface area-to-volume ratio of 3.75 ft^2/in^3, and should not be operated at pressure differentials above 600 psi. The research modules evaluated during the RO investigations (see Appendix A) demonstrated a water permeation rate of 0.057 $gal/ft^2/day$ when operated at 600 psi and temperatures near 75°F. From this data it could be concluded that the required volume of material is:

$$V = \frac{316.8 \times 0.95}{3.75 \times 0.057 \times 8.33} = 168.5 \text{ in}^3$$

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*Private communication with DuPont representative.

C H E M T R I C
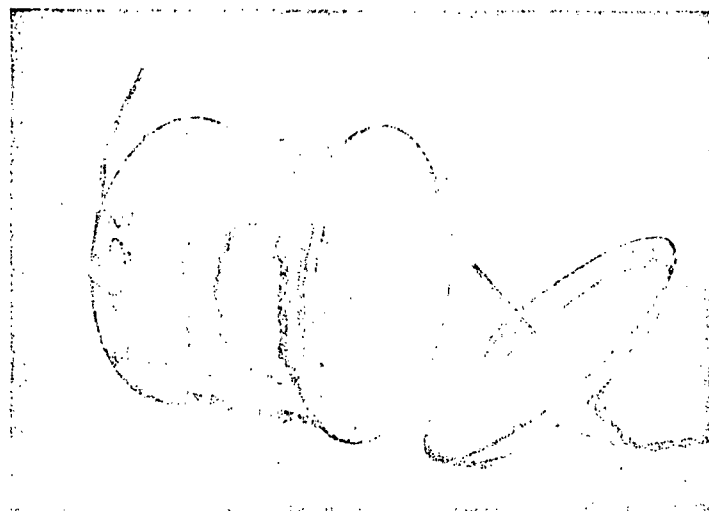
• EACH OF TWO SIDES IS 120 MAN-DAY CAPACITY.

• SHOWN WITH WICKS REMOVED & AFTER MISSION TEST.

• BAFFLES WERE DEFORMED DURING WICK-REMOVAL OPERATION.

• NOTE PERFORATIONS IN BAFFLES

Figure 9   FULL-SCALE SOLIDS DRYER

USED WATER
TANK

AgCl
COLUMN

POST TREAT
CELLS

PERMEATE

PERMASEP®
RO CELL

TO WASH
STATIONS

RELIEF

CONSTANT FLOW
REGULATOR

BRINE

FEED

BRINE

COARSE FILTER
FINE FILTER

HIGH-PRESS. PUMP

ACCUMULATOR
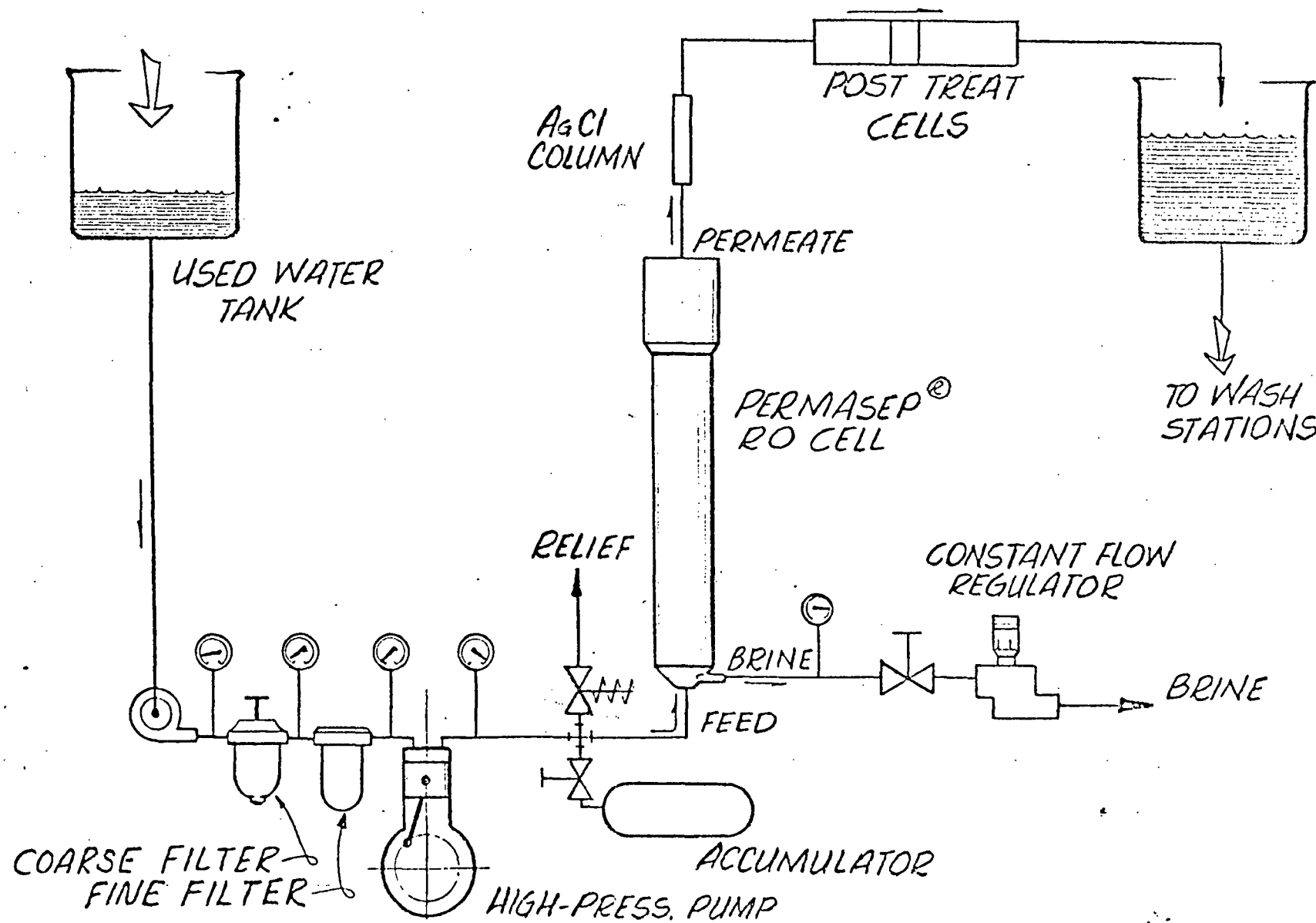
Figure 10   REVERSE OSMOSIS TEST SET-UP

However, if this size module was operated with a fixed feed and the ambient temperature dropped to 60°F (The lowest cabin temperature anticipated on a modular space station) the feed pressure required to maintain the desired permeation rate would exceed 600 psi. To avoid this problem and maintain a uniform water quality, the material volume (or surface area) must be selected so that the feed pressure rises to only 600 psi whenever cabin temperature falls to 60°F.

The water permeability of DuPont's B-5 Permasep material reportedly decreases 3% with every 1°C decrease in water temperature. Therefore, at 60°F the water permeability should be only

$$0.057 \times (1 - .03 \times 15/1.8), \text{ or } 0.0426 \text{ gal/ft}^2/\text{day},$$

and the minimum volume required should be

$$168.5 \times 0.057/0.0426, \text{ or } \underline{\underline{225 \text{ in}^3}}$$

Since the research modules have an internal volume less than $0.5^2 \ \pi/4 \times 48$, or 9.4 in^3, a substantially larger module was required to avoid plumbing more than 24 research modules in parallel. DuPont would not consider fabricating a special size cell for this program; however, they happened to have on hand a surplus module which has an internal volume of $4^2 \ \pi/4 \times 36$, or 452 in^3. Since the actual volume of material in this module is in the range of 300 to 350 in^3, it was decided to purchase this module. Figures 11 and 12 show a photograph and cut-away drawing of this module.

Feed entering the purchased module is distributed by a centrally-located porous tube so that it flows radially across the outer surface of the hollow nylon fibers. Permeate is collected at one end of the U-shaped fiber bundle, while brine can be withdrawn from the feed end or the two side-arm outlets on the cylindrical shell.

3.3.2 Feed Pump

A search of available high-pressure water pumps indicated that Hills-McCanna Model MA11-332-D Metering and Proportioning Pump was the best pump available for evaluating the selected DuPont Module. With a 1/3-HP electric motor, this pump delivers 3 gph against a 600 psi backpressure.
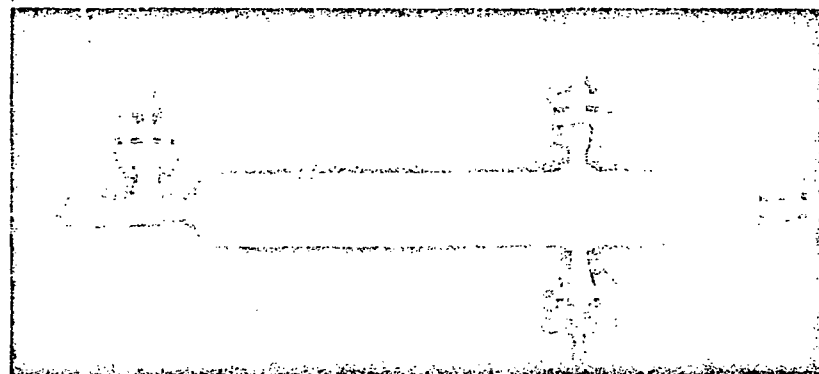
Figure 11   PHOTOGRAPH OF PERMASEP UNIT
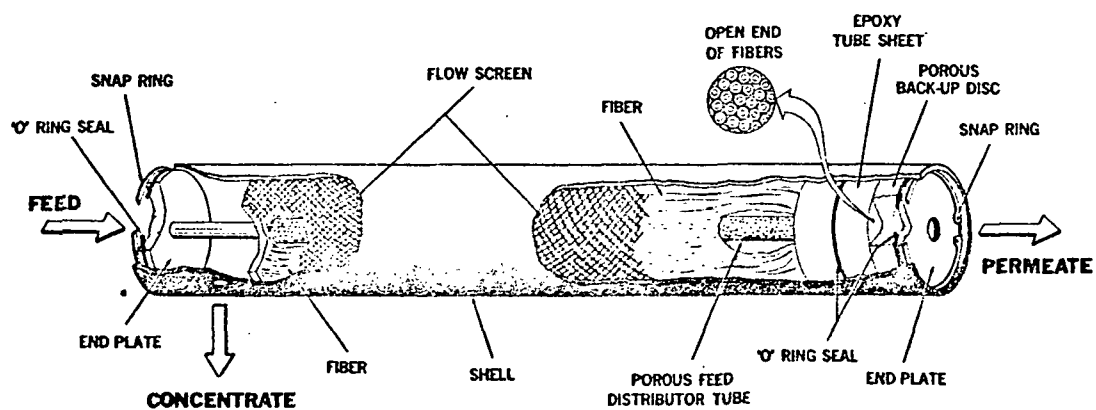
Figure 12   CUT-AWAY DRAWING OF PERMASEP UNIT

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3.3.3 Bleed Valve

The constant flow regulator shown in Figure 10 is a Brooks Model 8840SS Flow Controller - which is capable of maintaining brine bleed-off rates in the range of 0.5 to 400 cc/min, with pressure differentials up to 500 psi. This regulator was selected because the design bleed rate is,

$$\frac{3.6.8 \times 0.05 \times 454}{60 \times 24}, \text{ or } 4.83 \text{ cc/min,}$$

and the normal feed pressure anticipated during laboratory testing was less than 450 psi. This regulator was replaced by a micrometer-type needle whenever feed pressure did exceed 500 psi.

3.3.4 Prefilters

Several types of prefilters were used during the test program. Initially, two flushable Ronningen & Petter (Dover Corporation) Model SS-1112-STD filters were used - one containing a 700 mesh stainless steel screen, and the other, a 1 - 3 micron polypropylene fabric. When it was discovered that back-flushing could not be adequately performed, with the water pressure and quantity available, these filters were replaced. Cuno's (AMF) Model GH Auto-Klean Filter (ca. 88μ) was selected for the coarse filter, and their Model 1M1-40069-01 Micro-Klean Filter with a 3μ in-depth filter cartridge was selected for the fine filter.

CALIBRATION TESTS

The new compression distillation unit, and the Permasep RO Module purchased from DuPont, were subjected to a series of checkout and calibration tests prior to starting the first 30-day performance test. The results of these tests, and the few modifications necessary to maximize performance, are described in the following subsections.

4.1 Distillation unit

The checkout tests and most of the calibration tests were performed with deionized tap water. A series of calibration tests with pretreatment solution added to deionized water were also made to determine any effects on performance. The calibration test data were used later in this program for comparison to recalibration test data - to determine if system performance had degraded as the result of extensive testing with real wastes.

4.1.1 Checkout Tests

Six checkout tests, ranging in duration from 3-1/2 to 8 hours, were performed over a 15-day period to identify and rectify any electromechanical problems.

The first problem identified was excessive noise and high power consumption. The problem at first was thought to be caused by excessive circumferential clearance in the high-speed (3200 rpm) motor-to-compressor coupling - because the coupling included an intermediate body which compensated for any misalignment between the driving and driven members. It was reasoned that the intermediate member was resonating in circumferential vibration, and that a coupling which did not have circumferential freedom should be used. Several couplings of this type were installed, but the noise persisted. Finally, the problem was solved when another problem was identified and corrected.

After each checkout test the unit was partially disassembled for inspection. It was noticed that the pregreased timing gears were dry after several hours of running. From this observation it was concluded that the excessive backlash was causing the noise problem and overheating of the gears. One of the gears was then plated with electroless nickel to reduce the backlash from 0.004 to 0.002 inches. This solved the noise, coupling and gear lubrication problem - as evidenced by subsequently running these gears for more than 2000 hours without any visible evidence of wear.

The first checkout test indicated that the recovery rate was lower than it should have been at the operating conditions.

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However, the rate increased each time the unit was tested. On inspection, it was determined that some of the grease injected to lubricate the four compressor bearings had been diverted into the lobe housing, and decreased the internal clearances. The end clearances were then reduced (by machining the housing) from 0.012 inches to 0.006 inches, so that higher volumetric efficiencies could be obtained without grease. Also, the lubrication frequency was reduced to avoid the build-up of excessive amounts of grease in the compressor. Later in the program this compressor was replaced with one which does not require any grease to achieve high volumetric efficiencies.

Another problem which occurred during the checkout tests was failure of the cogged-belt rotating the bowls. On inspection it was discovered that the small pulley had been improperly machined. A new pulley was fabricated and the belt was replaced with an O-ring - which is more capable of withstanding eccentricities and wobble than a cogged belt.

No other significant changes were made during the checkout tests. Minor adjustments made were concerned with (1) reshaping the pick-up tubes to maximize pumping capacity, (2) adjusting the internal relief valve "cracking" pressure, and (3) adjusting the high-level sensor.

4.1.2   Calibration tests

Thirty-nine calibration tests, ranging in duration from 2 to 13 hours, were performed at various ambient temperatures to determine the thermodynamic performance of the distillation unit - using deionized tap water with and without pretreatment chemical, as the feed solution. This relatively large number of calibration tests were necessary because (1) several adjustments and corrections had to be made to achieve design performance, and (2) it was discovered that excessive quantities of "fresh" pretreatment chemical grossly affected the distillation rate.

The pretreatment chemical solution used contains an iodophor which has an iodine vapor pressure near 1 torr at room temperature. Initial calibration tests were performed with feed solutions which contained up to 5% pretreatment chemicals, without recycling of the excess feed. Under these conditions the distillation unit acted like it had "sprung" a leak; i.e., the condenser purge valve had to be full open to avoid excessive pressures and motor power. However, when untreated water was used as the feed the operation returned to normal. From this it was concluded that iodine was being "stripped" from the feed solution faster than it could be removed by normal purging. The pretreatment concentration was then reduced to normal levels

(ca. 1%) and the recycle loop used, so that the rate of iodine evolution was substantially less than the purge rate necessary to maintain the partial pressure of non-condensibles at an insignificant level in the condenser.

Other problems encountered were (1) premature failure of the 65-watt drive motor, and (2) a cracked weld between the evaporator and condensers. The drive motor failed due to the excessive running temperatures encountered when other problems had caused an increase in motor power. Since only two 85-watt motors were on-hand, it was then decided to weld a water cooling coil around the vacuum-tight housing in which the motor was press-fitted. Extended heat transfer surfaces (fins) could have been used - but it was more expediant to use water cooling at this stage in the program.

At this point recovery rates could not be reliably reproduced. The unit was disassembled for detailed inspection. It was then discovered that several spot welds, in a lap-joint between the evaporator and condenser, had broken to allow vapor leakage from the condenser to the evaporator. After these welds were repaired the recovery rates were reproducible - but still lower than they should have been at the operating conditions.

Finally, it was concluded that the slip speed was higher than predicted. The internal clearances in the compressor were then reduced by coating the end plates with Texaco's Uni-Temp 500, the same lubricant used in the compressor bearings. This adjustment produced the desired performance, as indicated in Figure 13. Later in the program this same performance was achieved with another compressor which used a "paint-like" coating to minimize internal clearances.

All of the calibration data was obtained with the distillation unit enclosed in a sheet metal cabinet - as it would be installed in a modular space station. Without forced circulation within the cabinet the cabinet temperature averaged 10°F above the laboratory (ambient) air temperature, and the condenser saturation temperature averaged 20°F above the ambient temperature. Therefore, the condenser temperature of interest ranged from 80 to 100°F, instead of the 60 to 80°F range assumed for design analysis (Section 2.2). Higher than normal cabinet temperatures were produced by turning on a light bulb inside the cabinet, while lower than normal temperatures were simulated by ventilating the cabinet with ambient air. The test data are presented as horizontal lines because recovery rates were averaged for periods of several hours, during which time the condenser temperature would vary as the result of starting transients and/or variations in the ambient temperature.
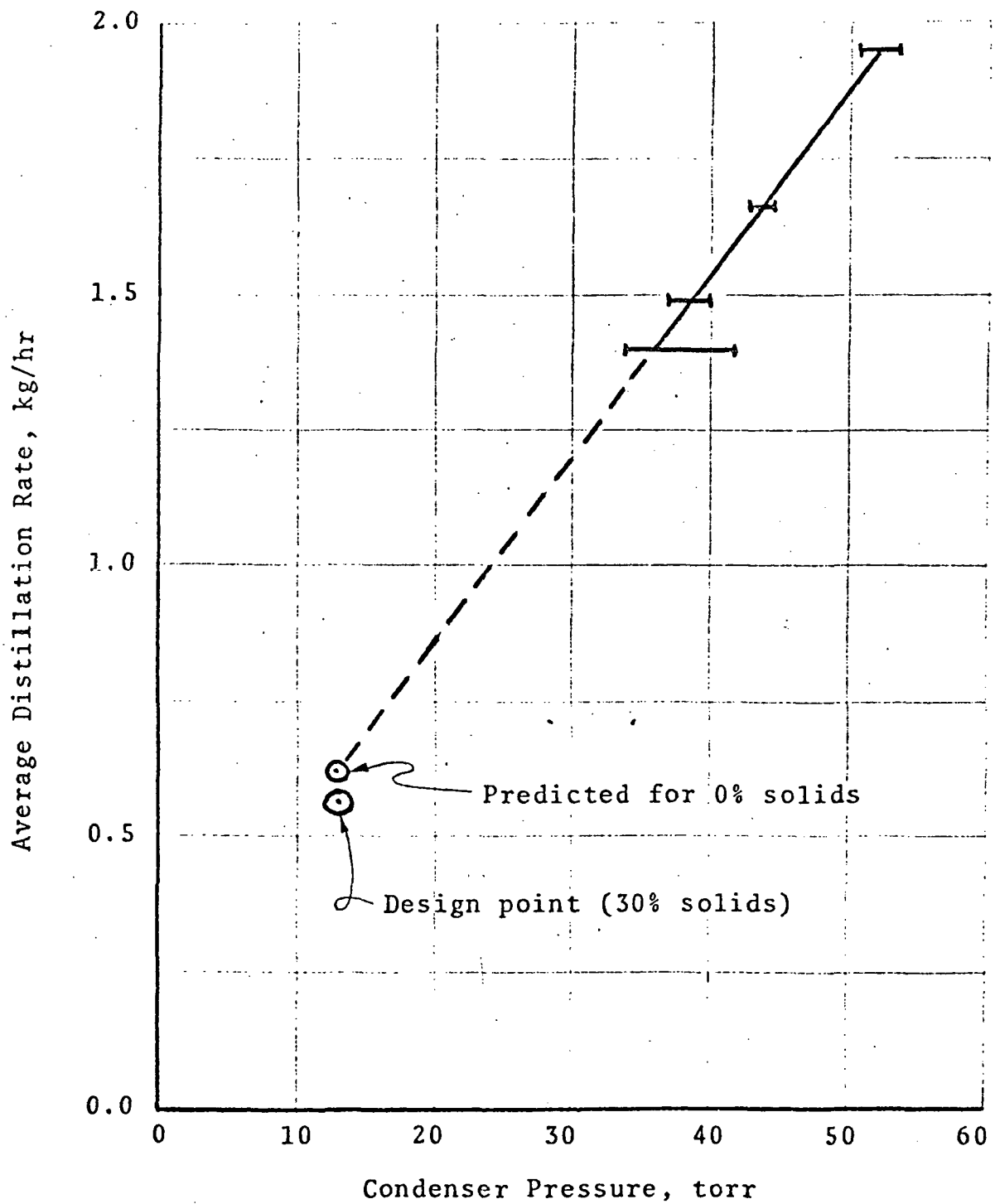
Figure 13   DISTILLATION UNIT CALIBRATION

C   H   E   M   T   R   I   C

Figure 13 shows that the valid calibration points can be correlated by a straight line, which can be extended to intersect the predicted performance at 60°F, when the feed contains 0% solids. Since variations in recovery rate should be primarily a function of vapor density, and vapor density is essentially a linear function of vapor pressure, it was concluded at this time that the distillation unit was performing as designed - and that tests with real waste should be initiated. As shown later in this report, compressor efficiency was actually lower than predicted, but this was offset by higher than anticipated heat transfer coefficients when operating with solids concentrations less than 50%.

The motor power required to perform these calibration tests ranged from 95 watts at P_c = 35 torr, to 112 watts at P_c = 54 torr. Since the corresponding recovery rates were 1.4 kg/hr and 1.95 kg/hr - the specific energy demand ranged from 68 to 58 watt-hr/kg (31 to 26 watt-hr/lb) Thus, the specific energy demand for this unit is substantially less than for any previously developed compression distillation unit.

4.2 RO Module

The Permasep Module was calibrated with deionized tap water - using the feed pump and pressure gages described in Section 3.3, and a graduated cylinder to measure the permeate flow rate over specific time periods. The pump stroke was adjusted to vary the feed rate, and thereby, determine permeate flow rate as a function of feed pressure.

Three calibration runs were performed - namely, one at 5.7 kg/hr (the design rate) and two at off-design flow rates. Figure 14 shows that at 5.7 kg/hr the resultant cell pressure was as predicted if the module contains 300 in^3 of B-5 Permasep material (i.e., P = 600 x 168.5/300, or 337 psi). However, at 3.26 kg/hr the resultant cell pressure was as predicted for 350 in^2 of material - while at 9.65 kg/hr it was higher than predicted for 300 in^3 of Permasep material. This non-linear result suggests that the water permeability of this material is a function of pressure as well as temperature; which is understandable when it is considered that compressive loading of the hollow fibers should compact the wall. However, the following correlation equation shows that the pressure affect is relatively small.

$$\dot{w} = 0.033 \ (\Delta P)^{0.9}$$

where $\dot{w}$ = permeation rate, kg/hr

P = pressure potential, psi

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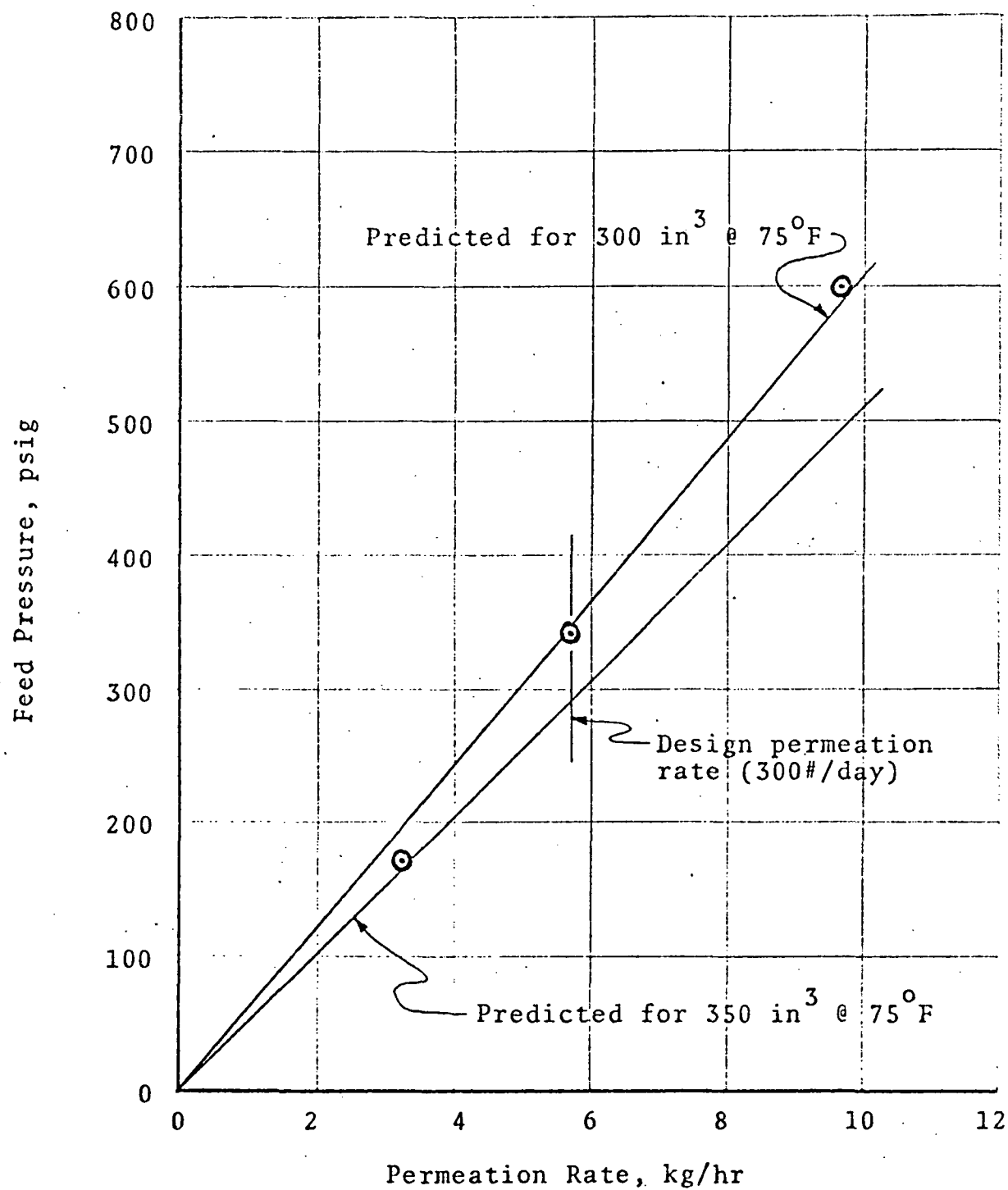
Figure 14   PERMASEP MODULE CALIBRATION

4-6

C   H   E   M   T   R   I   C

The primary objective of this test was to demonstrate that a compression distillation unit can process the urine and urinal flush water produced by four men over a 30-day period, without any malfunctions. Secondary objectives were to redemonstrate that compression distillation can produce potable water - with a specific energy demand less than 55 watt-hours per pound, and a water yield near 98%. These objectives were accomplished, as evidenced by the following summary of the work performed.

5.1 Test Set-up & Procedure

Preparation for the 30-day urine-loop test required the addition of several components to the set-up used for calibrating the distillation unit. The final arrangement of components is illustrated in Figure 15.

The dryer was placed at a branch in the recycle loop immediately downstream of the recycle pump to avoid feeding fresh waste water into the dryer. A needle valve was located in the branch line to control the rate at which recycle liquor was bled to the solids dryer. Output vapor was passed through a passive condenser and a gravity trap, which was purged through the evaporator. The dryer condensate could have been delivered directly to the evaporator, but the trap was used to permit measurement of condensate quantity and quality.

The original double-bladder tank, built by Marquardt, was placed in the set-up to accummulate flush water (AgCl-dosed condensate) and waste water. This tank was modified by the addition of another port so that waste water and/or recycle liquor does not stagnate in the waste side of this tank.

The solids filter located in the recycle loop was a Cuno Engineering Corporation Type 1M plastic housing, with a Micro-Wynd II cartridge. Initially, a 5-micron cartridge was used; after 15 days it was replaced with a 25-micron cartridge.

The post-treatment components include the biological filter (BF), activated carbon filter (ACF), a silver chloride column (AgCl) and the deionizer (I-X). Their description is as follows.

BF: Pall Trinity Micro Corporation Cartridge #ACF4463W (0.35 microns absolute) in a model #MDG4463G4Z3 housing.

ACF: A 1-1/2" stainless steel pipe, with screw-on end caps, to provide a 1.5" x 12.0" bed of Barneby-Cheney type 365 (20 x 50 mesh) activated carbon.

5-1
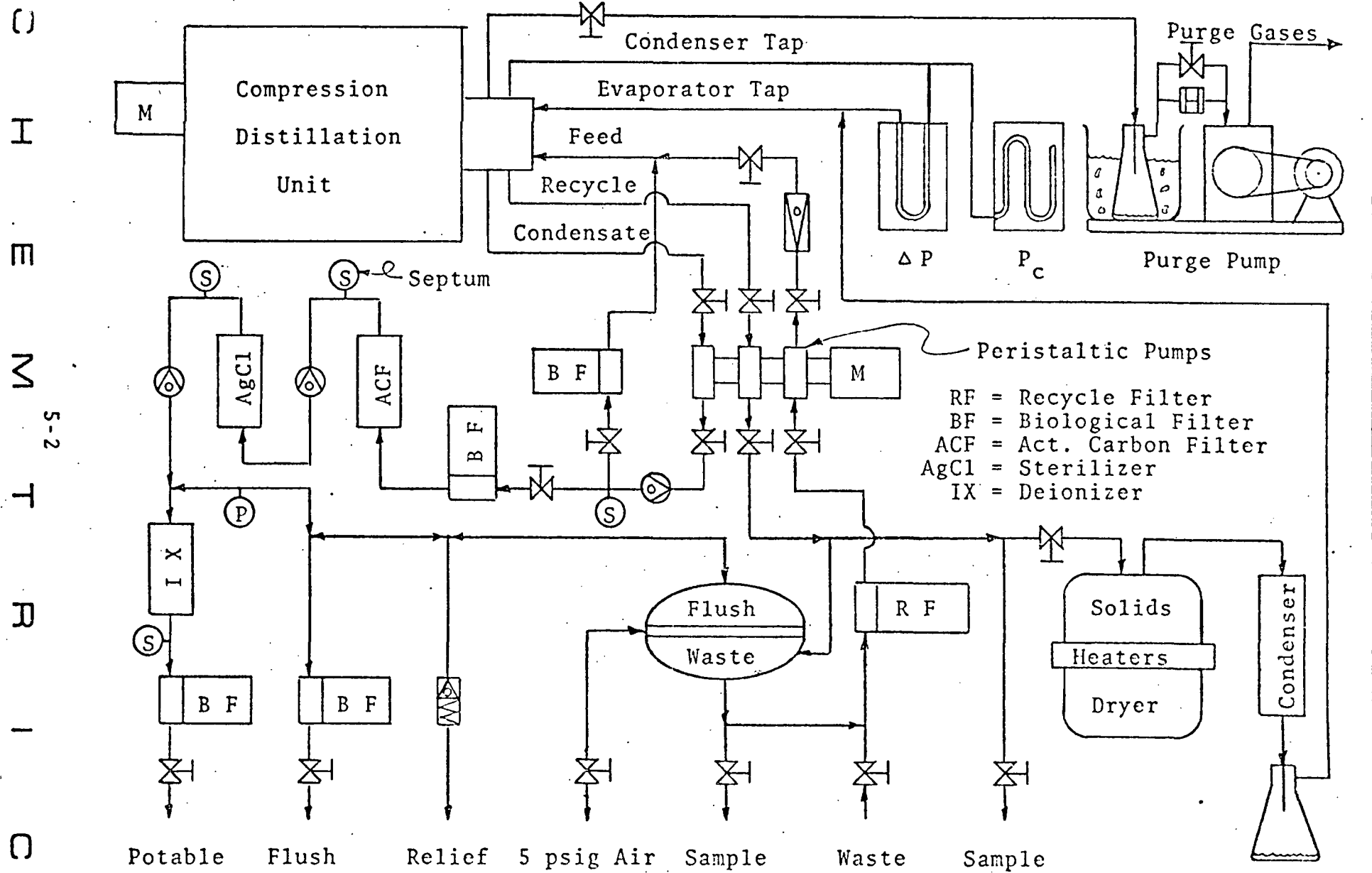
Figure 15   FLOW SCHEMATIC FOR 30-DAY URINE-LOOP TEST

AgCl: PVC pipe, with end caps, fabricated under contract NAS9-5119 and packed with 165 grams of AgCl particles and 205 grams of glass beads.

I-X: Stainless steel canister fabricated under Contract NAS9-5119 - and packed by CHEMTRIC with 26 in^3 of Amberlite IR-120 cationic resin plus 26 in^3 of Amberlite IR-45 anionic resin.

The ACF and I-X were sized to treat the raw condensate distilled from the urine produced and the pretreated flush water used by 4 men over a 30-day period; no provisions were made for the removal of trace contaminants which may carry over when humidity condensate is processed with urine and urinal flush water. The AgCl column was assumed to have an indefinite life. Based upon Pall's data, the BF was expected to be effective for about 10 days.

Urine and urinal flush water were collected for this test to closely simulate actual conditions onboard a modular space station. First, it was assumed that the average number of micturations would be 6 per man-day - and that the quantity of pretreatment solution and flush water used for each micturation would be the same, regardless of the quantity of urine voided during each micturation. Also, it was assumed that the resultant mixture may be frequently stored at ambient temperature for periods greater than 24 hours. Therefore, each day 24 one-liter plastic bottles were loaded with 150 grams of flush water plus 5 grams of pretreatment solution, and distributed to CHEMTRIC employees for collecting their own urine at work and/or at home. The used bottles were then returned to the laboratory and used as required to feed the urine loop during that day or the next day. Thus, the number of micturations was controlled instead of the urine quantity, and the treated urine frequently was stored at room temperature for periods of more than 24 hours.

The pretreatment solution added to each collection bottle during this test was:

Iodophor	2.45	gms
Sulfuric Acid	0.34	
Antifoam	0.13	
Water	2.08	
	5.00	gms

A detail discussion of the reasons for selecting this formulation is presented in Section 7.

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The procedure used each day in operating the urine loop for 30-days was:

1. Transfer the contents of several collection bottles into the waste tank.

2. Energize the distillation unit and pump assembly drive motors.

3. Add more treated urine to the waste tank whenever the compound gage indicated that the waste tank was empty (P < 5 psig).

4. Periodically draw-off condensate; first, as flush water until 3600 ml was available for preparing the next day's collection bottles, and then, the remainder as potable water.

5. After the contents of 24 collection bottles had been added, and the waste tank was empty, and the delta P started to increase (indicating a dry evaporator) - shut-off the drive motors.

6. Measure the quantity of condensate in the dryer trap, and transfer it to the waste tank for processing the next day.

Adjustments made during the test were limited to:

1. Trimming the purge valve setting to minimize purge losses.

2. Trimming the dryer feed valve to maintain the recycle liquor solids concentration near 30%.

The measurements made included:

1. Net weight of each collection bottle.

2. Weight of flush and/or potable water produced every half hour.

3. Power to the distillation unit's drive motor.

4. Condenser pressure.

5. Condenser-evaporator delta P.

6. Dryer temperature at different locations.

7. Weight of condensate in traps.

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Water samples were withdrawn each day, through septums at several locations, to determine ammonia concentration, COD, pH, specific resistance, sterility, aerobic count, coliform count and yeast and mold count. In addition, potable water samples were periodically submitted to the NASA MSC for chemical analysis. Standard methods were used for the ammonia, COD, pH and resistance analyses.

The membrane filter technique was used in performing all biological counts. This entailed filtering 100 ml of the respective sample through a 0.45μ millipore filter followed by 300 ml of sterile water. The membrane filter was then placed on agar media; following placement on the agar plate, the plate was sealed in a plastic container and incubated at 35°C for 48 hours. Tryptone Glucose Extract agar, Endo agar, and Rose Bengal-Malt Extract agar were used for aerobic count, coliform count, and yeast/mold count, respectively. The initial counts were performed with undiluted samples; subsequent tests were conducted with 1:10 dilutions of the sample.

Sterility testing was performed by innoculating 5 tubes of thioglycolate broth - 135C (BBL). The tubes were incubated at 35°C and inspected at 24 and 48 hours for turbidity.

5.2 Results & Discussion

The urine loop was operated for thirty consecutive days - plus three days for a special experiment, and two more days for recalibration of the distillation unit. The daily operating time ranged from 4-1/2 to 9-1/2 hours, depending upon the quantity of urine collected and the concentration of dissolved solids in the recycle liquor. The distillation unit did not experience any failures, nor require any maintenance. Planned maintenance on the urine-loop included changing the biological filter and the activated carbon filter. Unplanned maintenance required was limited to (1) changing the silicone tubing in the recycle pump, which periodically took a permanent set in the collapsed position, and (2) replacing the solids filter.

5.2.1 Thermodynamic Performance

The daily inputs and outputs for the 30-day test are summarized in Table 1. The results obtained, and the anomalies and trends in the data are described as follows.

1. The daily input ranged from 6,323 grams to 15,274 grams depending upon the quantity of urine collected. Since 155 x 24 x 30, or 111,600 grams of the total input was treated flush water, the actual quantity of urine processed was 310,763 - 111,600, or 199,163 grams. Thus, the average mass of urine collected was 199,163/24 x 30, or 277 grams/micturation.

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Table 1   PERFORMANCE SUMMARY FOR 30-DAY URINE-LOOP TEST

Day No.	Total Input (grams)	Total Output (grams)	Purge Loss (grams)	Dryer Output (grams)	Recov. Rate (kg/hr)	Specific Energy (W-H/kg)
1	10,478	9,302	128	0	1.52	66
2	15,274	14,249	160	0	1.51	64
3	10,327	10,278	212	0	1.35	64
4	10,743	10,494	120	0	1.28	77
5	10,810	9,633	94	0	0.93	105
6	10,251	9,855	74	640	1.29	75
7	10,379	9,771	109	210	1.45	68
8	10,357	10,181	179	539	1.18	85
9	10,328	9,527	120	393	1.41	68
10	10,260	10,095	210	184	1.25	81
11	13,019	12,444	220	245	1.35	77
12	10,272	9,246	125	125	1.20	85
13	10,155	8,738	169	260	1.34	77
14	8,274	7,745	125	298	1.35	77
15	6,323	5,179	108	204	1.31	77
16	7,939	7,421	41	268	1.35	73
17	9,091	8,102	36	287	1.34	70
18	11,708	11,091	78	389	1.35	70
19	10,923	10,114	86	437	1.48	66
20	8,799	8,586	129	370	1.58	66
21	10,142	8,965	72	443	1.27	77
22	10,552	10,041	100	476	1.32	75
23	9,867	9,751	100	295	1.33	70
24	11,307	9,947	57	425	1.27	75
25	10,715	10,618	50	633	1.28	73
26	11,293	10,843	91	320	1.30	73
27	11,142	9,858	135	286	1.28	79
28	10,663	10,120	102	413	1.29	75
29	10,557	9,606	80	539	1.26	73
30	8,815	9,668	86	604	1.25	77
	310,763	291,468	3,396	9,283		

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2. The output was distributed as 150 x 24 x 30, or 108,000 grams of flush water with the reaminder as potable water and water samples.

3. Daily purge losses varied considerably due to "over trimming" the purge valve; however, the average loss was only 3,396 x 100/310,763, or 1.09% (based on total mass input).

4. Recycle liquor was not fed to the dryer until day #6, to obtain a rapid build-up of solids in the recycle loop. Near the end of day #5 it was determined that the concentration of dissolved solids in the recycle loop had exceeded 30%. The dryer's daily output (condensate in gravity trap) varied considerably because of "over trimming" the dryer feed valve.

5. Recovery rate decreased, as expected, as solids concentration increased - and oppositely so whenever too much recycle liquor was bled-off to the dryer. On days 19 and 20 tap water was substituted for part of the feed due to a lack of sufficient urine; consequently the recycle loop was diluted and the recovery rate increased substantially.

6. As expected, specific energy essentially varied inversely with recovery rate.

The exact water yield could not be determined for this test, as done later in the program, because only the total mass in each collection bottle was determined. On a total mass basis, the yield was

$$\frac{291,468}{310,126} \times 100, \text{ or } 94\%$$

Since the concentration of dissolved solids in the feed should have been near 3%, the estimated water yield is 94 x 100/97, or 97%.

At the conclusion of the test the solids dryer was maintained in the dry-out mode for another 10 days. An additional 3,826 grams of condensate was recovered. Thus, if this water had been returned to the recycle loop during the test, and the input solids was 3% of the feed, the water yield would have been over 98%. However, this result indicates that the dryer is not capable of achieving the instantaneous drying rate necessary to achieve yields near 98% when the loop processing rate is near 1.4 kg/hr.; more heat transfer area and/or more vapor passageway area is necessary to match the performance of the distillation unit.

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C   H   E   M   T   R   I   C

A significant result of this test is the fact that the estimated water yield of the distillation unit alone was substantially higher than anticipated. The total input to the distillation unit was 310,126 + 9,238, or 319,409 grams. Therefore, it's mass yield and estimated water yield are, respectively:

$$\frac{291,468}{319,407} \times 100, \text{ or } 91.3\%$$

$$\frac{91.3}{97} \times 100, \text{ or } 94\%$$

Thus, the solids dryer only increased the water yield from approximately 94% to 97%.

During this test, and all subsequent tests, the condenser pressure was maintained in the range of 35 to 40 torr, by heating or ventilating the cabinet surrounding the distillation unit. After the concentration of dissolved solids in the recycle loop was stabilized around 30%, the delta P ranged from 8 to 10 torr, and the average was very near 9 torr. Thus, the heat transfer coefficient was very near 75 Btu/hr-ft^2-^{O}F, the value used in the preliminary design calculations presented in Section 2.2.3. This test indicated that the design analysis accurately predicted thermodynamic performance - at least when the solids concentration is near 30%.

5.2.2 Expendables Required

The expendable items used in the urine-loop were:

A. Pretreatment Solution (PS)
B. Recycle Filter (RF)
C. Biological Filter (BF)
D. Activated Carbon Filter (ACF)
E. Silver Chloride Column (AgCl)
F. Deionizer (I-X)
G. Solids Dryer (SD)

The quantity of PS used over the 30-day period was 30 x 24 x 5, or 3600 grams - to treat 199,163 grams of urine. Thus, the percentage of PS used was 3600 x 100/199,163 or 1.8%. Since the formulation was 58% chemicals and 42% water (see page 5-3) the percentage of chemicals used was 1.0%. As shown later in this program (Section 7) this was more than necessary to stabilize the urine. However, this relatively large amount of PS was used to positively avoid any stabilization problems during the initial test series, whose primary objective was to demonstrate reliability.

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Initially, a 5-micron cartridge was used to filter the recycle liquor and the fresh waste water fed into the distillation unit. On day #10 the 5-micron filter plugged, as indicated by a vacuum reading on the compound gage, shortly after the waste tank was loaded. Since the distillation unit had not experienced any internal plugging, and 10 days seemed to be a relatively short useful life for a solids filter, the plugged 5-micron cartridge was replaced with a fresh 25-micron cartridge, which performed satisfactorily for the remainder of this test series. However, this was not a good test of the solids filter because a separation of the recycle liquor occurred in the filter, as observed through the transparent housing. A dark, more dense, layer settled at the bottom - while the lighter liquor passed through the upper portion of the cartridge. Therefore, gravity may have aided in prolonging the useful life of this filter.

The biological filter (BF) and the activated carbon filter (ACF) were replaced at the end of days #10 and #20 - because previous tests performed under Contract NAS 9-9014 had shown that the useful life of the biological filter is on the order of 10 days, and the ACF was sized to process the raw condensate obtained from 40 man-days of urine and treated urinal flush water. During a later test the useful life of the BF was extended to at least 30 days.

The silver chloride column (AgCl) was not replaced because it has an indefinite life.* The deionizer (I-X) and the solids dryer (SD) were not replaced because they were sized for 120 man-days of operation.

5.2.3 Water Quality

As stated in Section 5.1, three types of water analyses were performed during the 30-day, urine-loop test - namely, daily analyses of key parameters, periodic biological analyses and periodic detail analyses of the product water. The results obtained are described as follows.

5.2.3.1 Daily Analyses

Table 2 contains the results of the daily water analyses performed at CHEMTRIC. The table is largely self-explanatory although some remarks are warranted about process

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* This particular column was fabricated by Melpar under NAS 9-5119, and previously used by AMGLO under Contract NAS 9-9014.

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Table 2   DAILY [CHEM] ANALYSIS FOR 30-DAY URINE LOOP TEST

Test Day	Process Stage	NH$_3$ (ppm)	COD (ppm)	pH	Resistivity (K ohms-cm)	Remarks
3	Condensate	4.4	48	6.8	45.00	Carbon Fines
	ACF Effluent	4.3	48	7.1	26.00	Present
	Flush Water	---	---	6.9	28.00	
	Potable	---	---	6.9	56.00	
4	Condensate	3.2	54	5.7	70.00	Carbon Fines
	ACF Effluent	5.0	99	7.0	29.00	Present
	Flush Water	---	---	7.0	30.00	
	Potable	---	---	7.0	28.20	
5	Condensate	2.2	138	5.2	48.00	Carbon Fines
	ACF Effluent	1.3	248	5.8	27.00	Present
	Flush Water	---	---	6.9	25.00	
	Potable	---	---	6.2	750.00	
6	Condensate	2.0	140	5.3	46.00	Carbon Fines
	ACF Effluent	1.3	96	6.8	28.00	Present
	Flush Water	---	---	7.0	28.00	
	Potable	---	---	6.3	752.00	
7	Condensate	1.0	97	4.5	37.80	Carbon Fines
	ACF Effluent	1.0	131	6.9	21.00	Present
	Flush Water	---	---	6.9	22.00	
	Potable	---	---	6.0	820.00	
8	Condensate	1.2	73	4.8	57.50	Carbon Fines
	ACF Effluent	1.0	48	6.7	36.00	Present
	Flush Water	---	---	6.8	37.20	
	Potable	---	---	5.8	700.00	
9	Condensate	2.2	385	5.2	31.80	Carbon Fines
	ACF Effluent	0.6	141	6.7	38.20	Present
	Flush Water	---	---	6.9	40.00	
	Potable	---	---	6.0	760.00	
10	Condensate	1.0	105	4.3	28.00	Carbon Fines
	ACF Effluent	1.0	100	6.7	26.80	Present
	Flush Water	---	---	6.9	25.50	
	Potable	---	---	6.0	610.00	
11	Condensate	1.0	113	4.4	30.50	Carbon Fines
	ACF Effluent	1.0	63	6.8	24.00	Present
	Flush Water	---	---	6.7	29.00	
	Potable	---	---	6.2	510.00	

C H E M T R I C

(Continuation of Table 2)

Test Day	Process Stage	NH₃ (ppm)	COD (ppm)	pH	Resistivity (K ohms-cm)	Remarks
12	Condensate	10.0	188	9.1	4.90	Dryer Purge
	ACF Effluent	10.0	97	6.8	4.90	Line Connected
	Flush Water	---	---	6.9	8.60	To Evaporator
	Potable	0.5	---	6.6	800.00	
14	Condensate	81.0	168	9.0	4.70	Carbon Fines
	ACF Effluent	71.0	160	8.9	3.10	Present
	Flush Water	---	---	8.9	2.80	
	Potable	0.5	---	5.9	730.00	
16	Condensate	---	472	---	---	Analytical Ef-
	ACF Effluent	---	112	---	---	fort Limited to
						COD Calibration
17	Condensate	100.0	165	9.2	2.00	Carbon Fines
	ACF Effluent	100.0	134	9.1	1.70	Present
	Flush Water	---	---	9.0	1.90	
	Potable	0.5	---	6.0	680.00	
	Dryer Condensate	120.0	250	8.2	0.01	
18	Condensate	70.0	198	8.9	3.10	Carbon Fines
	ACF Effluent	60.0	101	8.9	2.20	Present
	Flush Water	---	---	9.0	2.40	
	Potable	0.5	---	6.1	650.00	
	Dryer Condensate	130.0	360	8.3	0.01	
19	Condensate	70.0	96	7.8	11.50	Carbon Fines
	ACF Effluent	57.0	70	7.6	12.70	Present
	Flush Water	---	---	8.0	12.20	
	Potable	0.5	---	5.8	510.00	
	Dryer Condensate	---	248	8.4	0.12	
22	Condensate	85.0	74	9.1	3.80	Filtered Before
	ACF Effluent	76.0	72	8.7	3.20	Analysis
	Flush Water	---	---	8.6	3.80	
	Potable	0.5	---	5.9	340.00	
	Dryer Condensate	---	180	8.6	0.01	

C H E M T R I C

(Continuation of Table 2)

Test Day	Process Stage	NH$_3$ (ppm)	COD (ppm)	pH	Resistivity (K ohms-cm)	Remarks
23	Condensate	70.0	88	8.7	4.95	Filtered Before Analysis
	ACF Effluent	68.0	44	8.0	5.10	
	Flush Water	---	---	7.9	4.80	
	Potable	0.5	---	5.6	310.00	
	Dryer Condensate	---	260	8.3	0.01	
24	Condensate	89.0	120	8.9	3.90	Filtered Before Analysis
	ACF Effluent	84.0	44	8.4	4.10	
	Flush Water	---	---	8.3	4.10	
	Potable	0.5	---	5.4	210.00	
	Dryer Condensate	---	170	8.0	0.40	
25	Condensate	87.0	110	8.6	3.00	Filtered Before Analysis
	ACF Effluent	85.0	72	8.2	3.70	
	Flush Water	---	---	8.3	3.80	
	Potable	0.5	---	5.4	220.00	
	Dryer Condensate	---	628	8.0	0.20	
26	Condensate	78.0	112	8.4	6.40	Filtered Before Analysis
	ACF Effluent	75.0	44	8.7	5.20	
	Flush Water	---	---	8.6	4.30	
	Potable	0.5	---	5.2	150.00	
	Dryer Condensate	---	394	8.0	0.30	
30	Condensate	38.0	168	8.8	4.60	Filtered Before Analysis
	ACF Effluent	48.0	64	8.8	4.60	
	Flush Water	---	---	8.6	4.30	
	Potable	0.5	---	5.3	104.00	
	Dryer Condensate	420.0	360	8.0	0.90	

C H E M T R I C

stage identification. Condensate is raw product water as it exits the distillation unit. This water is then processed through a biological filter (0.35 micron absolute) and a columnar bed of activated charcoal; water exiting this bed is designated ACF effluent. This effluent is then processed through the bed of silver chloride particles; the AgCl column effluent is designated flush water. Flush water is stored in the bladder tank which supplies the water necessary for flushing the waste collectors or to feed the deionizer. Deionized flush water is the final product water; at this point (deionizer outlet) the water is of potable quality.

The water analyses reveal a significant impact of the solids dryer on raw condensate quality. During the first eleven days of system operation, the solids dryer was purged separately from the still. On day 12, the dryer purge line was connected to the evaporator so that dryer vapor and non-condensable gases were introduced into the evaporator. The pH of the raw condensate changed abruptly after the above connections were made. As can be seen in the table, the pH increased from a mildly acidic value to mildly alkaline. The primary cause of this jump in pH is due to the increased ammonia concentration (which increased from 20 to 30 times higher than previously seen). This ammonia increase also caused a depression in specific resistance. The COD values were also elevated but less dramatically. Analyses of the dryer condensate were undertaken on day 17 to verify the presumption; these analyses clearly indicate that the dryer vapor output contained large amounts of ammonia and organic substances.

The poor quality dryer condensate is attributed to the dryer operating temperature rather than failure of the pretreatment chemical. Thermal decomposition of urea can produce sufficient ammonia to neutralize the acid residual in the treated liquor; the neutralization results in pH which permits ammonia distillation. Similarly, the high temperature elevates the partial pressure exerted by volatile organic substances. It is most significant to note that COD values obtained from the dryer condensate were always measurably higher than the distillation unit condensate.

A persistent problem occurred in conducting the analysis - namely, the leakage of carbon fines from the ACF unit. Leakage was due to improper packing of the carbon beds during fabrication. The main effect of the leakage was to elevate the COD value of the ACF effluent. However, the problem was not identified immediately and extensive testing of the COD reagents coupled with testing for interferring substances was carried out. After the problem was identified, the analytical routine was modified to include membrane filtration prior to COD analysis. Microscopic inspection of the membranes filter confirmed the presence of carbon fines.

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The potable water as indicated by the specific resistance measurements was of very high quality throughout the·test except the first four days. The off quality water was attributed to the biological filter located between the deionizer outlet and the potable outlet valve. The filter cartridge was installed in the housing with cursory washing; this washing was inadequate and contaminants were leached out of the cartridge by the deionized (potable) water. Later experience with identical cartridges verified this presumption.

5.2.5.2 Biological Analyses

The logistics requirements entailed in conducting both chemical and biological analyses were under estimated; this necessitated making a decision regarding which analytical effort to pursue rigorously. In light of the potentially significant insight to system performance offered by the chemical analysis, the biological analysis efforts were de-emphasized. However, biological analyses were conducted as frequently as possible.

The results of the biological analyses are presented in Table 3. The table presents the analytical values obtained from water samples taken at various process stages on the indicated test day. All samples were acquired aseptically through septum-type sampling ports.

The biological analyses indicate that the recyle liquor was sterile; sterility can be attributed to both the iodine residual and the high solids content of the recycle liquor. The AgCl column also performed quite effectively since on days 18 and 30 the ACF effluent was contaminated while the flush water was sterile. The high contamination levels encountered in the potable water are attributed to bacteria already present in the deionizer rather than bacteria contained in the influent water; deionizer influent is flush water which was shown to be sterile. Previous attempts to sterilize a resin bed resulted in either resin degradation or incomplete sterilization. The bed used in this test was assembled from (1) steam sterilized hardware components and (2) resins extensively washed in filter-sterilized deionized water. The bacteria found in potable water were undoubtedly descendants of bacteria present in the ineffectively decontaminated resins or those bacteria introduced during assembly of the deionizer. In later tests this biological problem was overcome by utilizing ion-exchange materials which can be sterilized before use.

C   H   E   M   T   R   I   C

Table 3 BIOLOGICAL WATER ANALYSES FOR 30-DAY URINE LOOP TEST

Test Day	Process Stage	Aerobic Count No/100 ml	Coliform Count No/100 ml	Yeast & Mold Count No/100 ml	Sterility Test (+ = Ster.) (- = Nonster.)
3	Recycle Liquor	0	0	0	+
11	Potable	300	0	0	-
15	Recycle Liquor	0	0	0	+
18	ACF Effluent	300	0	0	-
	Flush Water	0	0	0	+
	Potable	3000	0	0	-
19	ACF Effluent	0	0	0	+
	Potable	3000	0	0	-
22	Recycle Liquor	0	0	0	+
	Potable	3000	0	0	-
23	Potable	3000	0	0	-
24	Potable	3000	0	0	-
30	Recycle Liquor	0	0	0	+
	ACF Effluent	10	1	0	-
	Flush Water	0	0	0	+
	Potable	3000	0	0	-

5.2.3.3 Detail Analyses

As previously stated, composite water samples were frozen and shipped to the NASA MSC for detail analysis in the Crew Systems Laboratory. As shown in Table 4, the potable water met, with only two exceptions, the purity requirements set by MSC Spec-C35. The water met all requirements set by the Ad Hoc Committee on Water Quality of the National Academy of Sciences - with the exception of arsenic and boron; analyses for these elements were not performed because their occurrence was unlikely in the water and because the analytical procedures for these elements required excessive sample quantities. Color and turbidity measurements were also made; no color was detected in any of the samples and in every case turbidity amounted to 0.50 turbidity units or less (1.0 unit is the acceptable limit).

The deviation from purity specifications are noted as follows:

(1) Product water total carbon increased during the test period, even though the daily analyses indicated no such trend. From this result it is concluded that the organic carbon measured was that carbon contained in microbial cells flushed out of the deionizer by the product water.

(2) Out of spec concentrations of lead were found in the water samples from day 19 to 21 and 22 to 24. The cause of this condition is obscure. There is no lead or lead containing alloys anywhere in the system. It is unlikely that the lead was introduced inadvertently to the water samples since considerable care was taken in processing the samples both at CHEMTRIC and at the MSC. A possible explanation is the presence of elements that interfere with the analysis. Certain metals, namely, iron and silver can interfere with lead analysis producing high results. This also occurred in lead analyses of water samples taken during later tests.

5.2.4 High Solids Tests

During the 30-day urine-loop tests the concentration of dissolved solids in the recycle liquor was first allowed to build-up to approximately 30%, and then maintained near that level by bleeding recycle liquor to the solids dryer. At the end of the test it was decided to operate the urine loop for several more days without bleeding-off any recycle liquor, to determine the effect of higher solids concentrations on the performance of the distillation unit.

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Table 4 DETAIL WATER ANALYSES FOR 30-DAY URINE-LOOP TEST

Quality Parameters	Concentration (ppm)							Specified Limits		
	Day 1&2	Day 6&7	Day 10&11	16-18	19-21	22-24	30	USPHS (ppm)	ADHOC (ppm)	MSC35 (ppm)
ABS	---	---	---	---	---	---	---	0.05	NS*	NS
Ammonia	0.10	0.10	0.10	0.10	0.10	0.10	0.10	NS	NS	NS
Arsenic	---	---	---	---	---	---	---	0.01	0.50	NS
Barium	0.50	0.50	0.50	0.50	0.50	0.50	0.50	1.0	2.0	NS
Boron	---	---	---	---	---	---	---	NS	5.0	NS
Cadmium	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.05	0.01
C(CHC13 ext.)	---	---	---	---	---	---	---	0.20	NS	NS
Chloride	0.30	0.30	0.30	0.30	0.30	2.0	1.0	(1)	450.0	NS
Chromium (+6)	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.05	0.05	0.05
Copper	0.05	0.05	0.05	0.05	0.05	0.05	0.05	1.0	3.0	1.0
Cyanide	---	---	---	---	---	---	---	0.01	NS	NS
Flouride	0.05	0.05	0.05	0.05	0.05	0.05	0.05	1.0	NS	NS
Iron	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.30	NS	0.30
Lead	0.02	0.02	0.02	0.02	1.00	0.20	0.02	0.05	0.20	0.05
Manganese	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	NS	0.05
Mercury	.005	.005	.005	.005	.005	.005	.005	NS	NS	0.005
Nickel	0.10	0.10	0.10	0.10	0.10	0.10	0.10	NS	NS	0.05
Nitrate	0.05	0.05	0.05	0.05	0.05	0.05	0.05	45.0	NS	NS
Nitrite	0.05	0.05	0.05	0.05	0.05	0.05	0.05	NS	NS	NS
Phenols	---	---	---	---	---	---	---	0.001	NS	NS
Selenium	---	---	---	---	---	---	---	0.01	NS	NS
Silver	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.50	0.05
Sulfate	1.0	1.0	1.0	1.0	1.0	1.0	1.0	(1)	250.0	NS
Zinc	0.03	0.03	0.03	0.03	0.03	0.03	0.03	5.0	NS	5.0
COD	---	---	---	---	---	---	---	NS	100.0	NS
Total Carbon	14.0	20.0	18.0	24.0	43.0	29.0	45.0	NS	NS	NS
Total Solids	8.0	8.0	4.0	8.0	6.0	7.0	11.0	500.0	1000.0	500.0

* Not Specified.

(1) Sum of chloride & sulfate should not exceed 250 ppm

Figure 16 shows that, as expected, recovery rate varies inversely with the concentration of dissolved solids. However, the relationship is essentially linear between 0% and 64% dissolved solids - while beyond 64% recovery rate is drastically reduced as the solids concentration is increased. The sharp "knee" in the curve cannot be explained by boiling point elevation alone; from the physical appearance of urine liquor containing 69% dissolved solids it is concluded that viscosity also affects the recovery rate attainable with the distillation unit. This conclusion is substantiated by the fact that when the concentration of dissolved solids exceeded 70%, the recycle pump was unable to withdraw liquor from the evaporator. This condition occurred near the end of the third day of high solids testing.

5.2.5 Recalibration Tests

On the 34th day of this test series the distillation unit was flushed-out and recalibrated using tap water as the feed. Even though the recycle flow had stopped on day 33, due to excessive viscosity, it restarted automatically on day 34 when tap water was fed into the evaporator. From this result it is concluded that if the evaporator liquor is too viscous, recycle flow is retarded at the weirs (holes) in the evaporator dam - causing the evaporator level to rise and contact the high level sensor; the evaporator contents, of course, are then easily diluted when flush water is fed into the evaporator.

The distillation unit was recalibrated at two condenser pressure levels - namely, 32 and 42 torr. The corresponding recovery rate, averaged over four to five hour periods, were 1.38 and 1.54 kg/hr. Since these two data points straddle the performance line drawn in Figure 13 (page 4-4), it is concluded that the performance of the distillation unit had not degraded after 33 days of operation with urine and treated flush water.

5.3 Conclusions & Recommendations

The 30-day, urine-loop test achieved it's primary objective - namely, "to demonstrate that a compression distillation unit can process the urine and urinal flush water produced by four men over a 30-day period, without any malfunctions". Secondary objectives which were achieved include:

1. Demonstrating a water yield of more than 97% when operating with an external solids dryer.

2. Demonstrating a specific energy demand of less than 40 watt-hours per pound (88 w-h/kg) when the water yield is more than 93%.
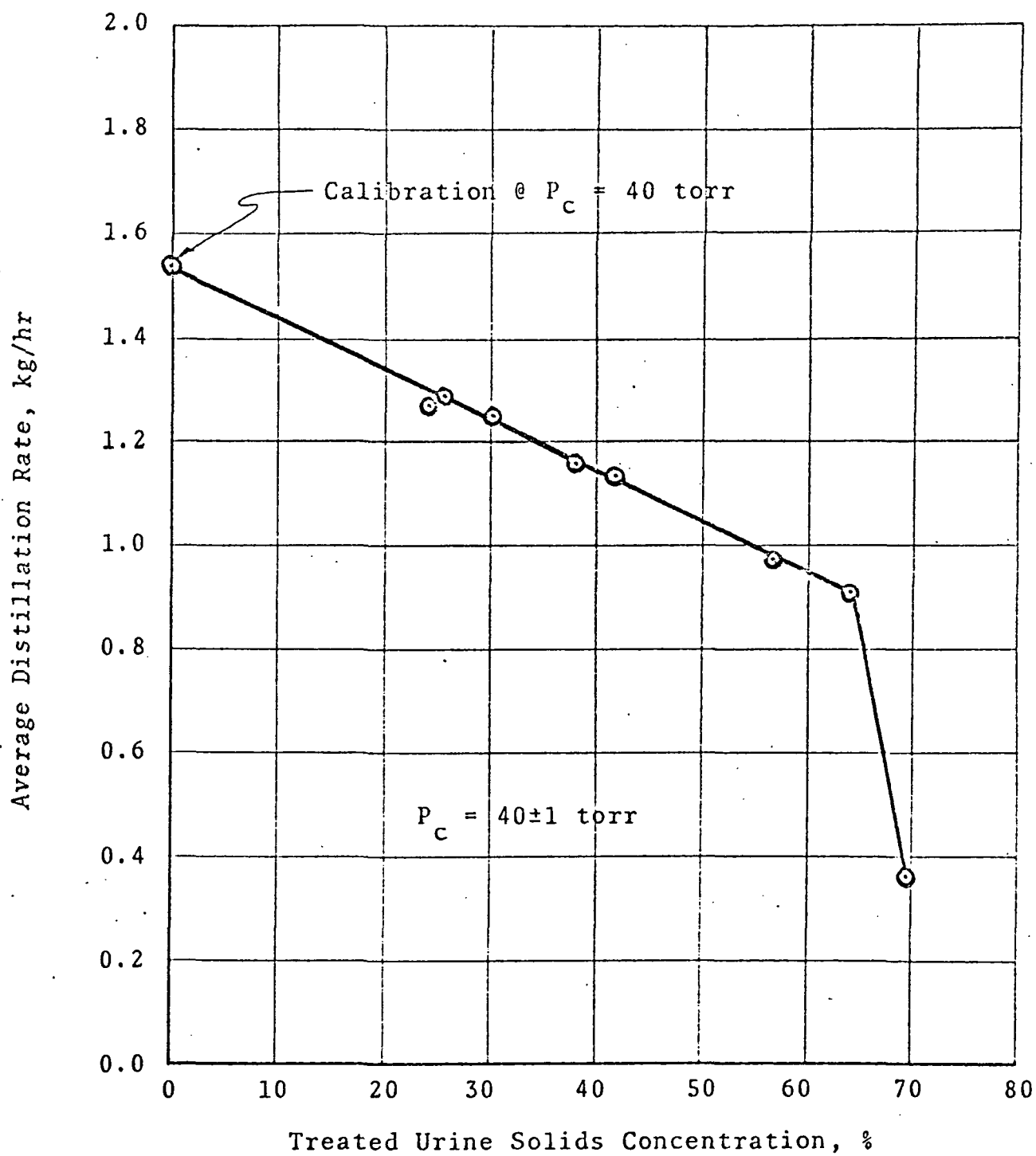
C H E M T R I C

Figure 16   EFFECT OF SOLIDS ON DISTILLATION RATE

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C H E M T R I C

Substantiating or useful design data obtained during this test include:

1. CHEMTRIC ~~~ atment chemical maintains the recycle liquor i~~~ le condition.

2. Saturating the filtered condensate with silver chloride ions maintains this water in a sterile condition.

3. The distillation unit can reliably process waste water and/or recycle liquor which has been passed through a 25-micron filter.

4. The average mass of urine voided during each micturation is more than 277 ml.

5. With a manually adjusted purge valve, the water lost through purging the distillation unit is only 1.09% of the waste water even though the distillation unit was started and stopped once each day, as would be done on a modular space station.

6. With a dissolved solids concentration of 30%, the average heat transfer coefficient is very near 75 B/hr-ft2-°F.

7. The distillation unit can reliably process concentrated, treated urine which has a dissolved solids concentration up to 64%.

8. The performance of the distillation unit does not degrade with time.

Unfortunately, this test did reveal that the following two problems must still be resolved.

1. Commercially-available peristaltic pumps, which are all designed for operation at gage pressures, do not yield the desired tubing life when used to withdraw recycle liquor and condensate from the distillation unit. A custom-designed pump is required to avoid collapse of the tubing when operating at subatmospheric pressures.

2. Sterile flush water passed through a non-sterilizable deionizer, to remove silver ions, becomes biologically contaminated. Later in this program this problem was solved by using a sterilizable ion-exchange resin.

C H E M T R I C

Another problem discovered, but also resolved during this test was the effect of solids dryer contaminants on quality of the raw condensate withdrawn from the distillation unit. Since the solids dryer increased the yield by only 3%, and the High Solids Tests demonstrated that the distillation unit can reliably process liquor containing up to 64% dissolved solids, it was concluded that yields over 97% could be achieved without a solids dryer by simply accummulating recycle liquor in a separate tank.

C H E M T R I C

30-DAY WASH-LOOP TEST

The primary objective of this test was to demonstrate reliable operation of a reverse osmosis (RO) subsystem processing used wash water, and feeding brine to a compression distillation subsystem - which also receives treated anal wash water from a two-stage fecal collector. The second objective was to determine the quality of the renovated wash water using a DuPont Permasep unit, and real wash water containing the surfactant and germicide recommended by the experiments reported in Appendix A. These objectives were accomplished and additional data was obtained, as evidenced by the following summary of the work performed.

Prior to starting this test the distillation unit was partially disassembled to inspect the compressor timing gears, which had been a problem area before the 30-day, urine-loop test. On inspection, it was found that each tooth was covered with a lubricant film, and no visual evidence of wear was observable. The gears were then relubricated before reassembly.

6.1 Test Set-Up and Procedure

Figure 17 on the next page illustrates the component arrangement used for the 30-day, wash-loop test. The significant features of this test set-up, and the reasons for it's arrangement, are described as follows.

1. The solids dryers used during the 30-day, urine-loop test was omitted, for the reasons stated in Section 5.3. Instead, a small portion of the recycle liquor was dumped each day. To avoid the need for more frequent dumping, the volume of the recycle loop was increased by installing an empty filter-cartridge housing in the recycle loop.

2. The deionizer was omitted because potable water was not required; the desired distillation product was filtered condensate saturated with silver-chloride ions.

3. A two-stage commode was not used because the unit furnished by Hamilton Standard for use on this program required some modifications, which could not be completed in time for this test. A two-stage commode was simulated by withdrawing flush water, dosing it with pretreatment chemical, and pumping it back into the waste tank.

4. Brine produced by the RO cell was measured and then pumped into the distillation waste tank - instead of being continuously forced into the recycle loop as would be done on a flight vehicle.

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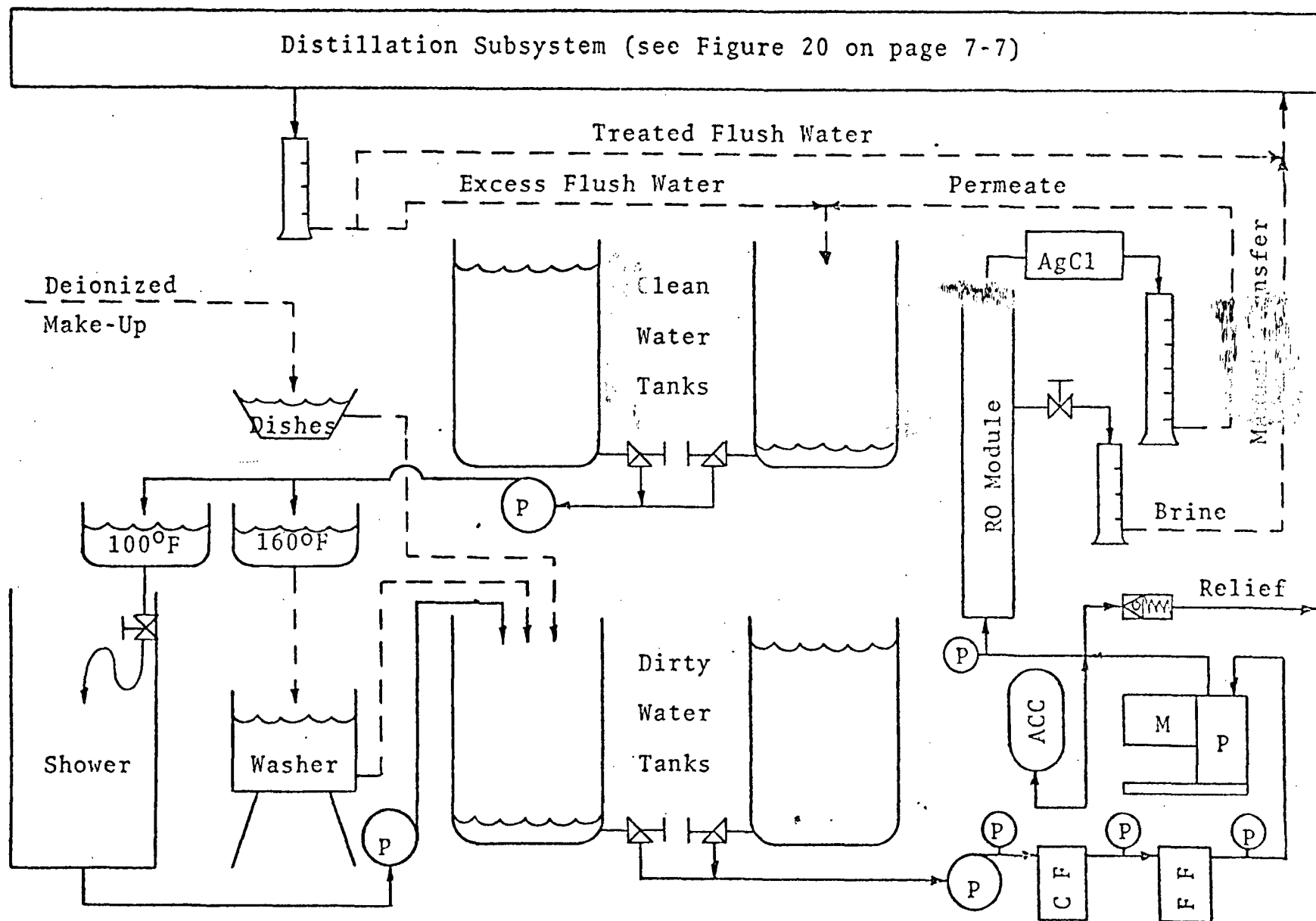
Figure 17   FLOW SCHEMATIC FOR 30-DAY WASH-LOOP TEST

5. Four polyethylene drums were used for holding renovated and used wash water, to avoid mixing and permit the accurate determination of integrated daily quantities.

6. Shower water and clothes wash water were pumped into graduated reservoirs, and heated to 100°F and 160°F, respectively, before use.

7. The quantity of used wash water collected was not measured - instead, the collection tank was topped-off each day to the same level with used dish water, and the quantity added was recorded as the make-up required to compensate for evaporation, purge and liquor-dump losses.

8. The wash water booster pump was necessary to compensate for pressure losses through the coarse and fine filters, and thereby, prevent cavitation in the RO feed pump.

9. Renovated wash water (RO permeate) was not charcoal filtered - because one objective of this test was to learn if using unfiltered permeate caused any significant dermatological problems.

10. The silver-ion sterilizer (the same one used in the 30-day, urine-loop test) was installed to assure the sterility of the renovated wash water.

11. Showering was accomplished in a sheet-metal stall - with a Tygon hose, sponge and cotton towel; a reciprocating-type washer, with a wringer, was used for clothes washing.

Real contaminants were obtained by showering members of the test crew, washing dirty dishes, and washing towels, socks and undergarments. The quantity and type of contaminants was uncontrolled. However, the quantity of wash water, surfactant and germicide used per man-day were as follows.

	Showers	Clothes	Dishes
Water (nom.)	7600 ml	1360 ml	400 ml+
Miranol C2M*	7.6 gm	1.36 g	0.4 gm
Hyamine 1622**	0.0	6.8 g	0.0
Anti-Foam	20 drops	0.0	0.0

* Solution containing 40% solids.

** Powder form

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Since one of the objectives of this test was to determine the quality of the renovated water, and permeate quality is a function of water yield, the RO yield was controlled at 70%, 85% and 95%, successively, during the test period. To avoid daily running times of more than eight hours, it was then decided to limit the amount of wash water to 1, 2 and 3 men/day, respectively. After the test was started, and it was determined that more brine could be distilled, the 3-man level was used for the 85% yield as well as the 95% yield tests.

6.2 Results & Discussion

The wash loop was operated for 30 consecutive days, to perform 35 days of wash water processing and three days of recalibration. Early in the test, two one-man-day tests (70% yield) could be performed within eight hours; therefore, this was done to maximize the amount of data obtained for the funds allocated. Fortunately, this deviation permitted five additional days of running at the end of the test, to evaluate gravitational effects on permeate quality.

During this test the distillation unit did not experience any failures; however, the feed rate had to be increased to maintain the recycle flow when the liquor became relatively viscous during the last 20 days of the test. Apparently, concentrated wash water containing 30% dissolved solids is more viscous than concentrated urine water containing the same amount of solids - and the feed-to-recycle ratio used for the urine-loop test was insufficient to avoid excessive solids concentrations (or viscosity) when processing concentrated wash water. Later in the program this problem was minimized by always flushing the evaporator clean with raw condensate before shutting-down the unit each day - to avoid the excessive concentrations experienced when the feed is exhausted each day, and the unit is subsequently "dried-out" prior to shut-down for the night.

Planned maintenance was limited to replacing the biological filter (BF) and activated carbon filter (ACF) in the distillation subsystem, at the end of days 10, 20 and 30.

Unplanned maintenance required during this test was:

1. Replacing the silicone tubing in the condensate pump during test day #1.

2. Repairing a leak in the AgCl column on day #5.

3. Replacing the silicone tubing in the distillation unit feed pump on day #6.

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4. Replacing the silicone tubing in the recycle pump during day #9.

5. Bleeding the RO feed pump on day #14, when it was gas locked.

6. Cleaning the RO prefilters on days #15 and #16, and replacing them with other types of filters on day #17.

Thus, the peristaltic pumps were most troublesome, as they also were during the 30-day, urine-loop test. Failure of the condensate and recycle pump tubing was anticipated, because in the commercial pumps used they eventually take a permanent set in the collapsed position when drawing against a vacuum. The feed tubing failure was unexpected, because it is always pressurized outwardly; since the failure was due to external abrasion by the peristaltic rollers, it is concluded that a flight-qualified peristaltic pump must have gear-driven rollers to minimize relative motion between the tubing and roller surfaces. Also, peristaltic pumps should be "canned", so that if the tubing ever does break in-flight, the leakage will be confined.

6.2.1   Thermodynamic Performance

Table 5, 6 and 7, respectively, summarize the daily inputs and outputs for the 70%, 85% and 95% RO yield tests. The results obtained, and the anomalies or trends in the data are described as follows.

6.2.1.1   70%-Yield Tests

These tests, which simulated the support of one man, were each of relatively short duration - as indicated by the running times (power-on) listed for the RO feed pump and the distillation unit drive motors. Consequently, two tests were performed each day.

The data obtained from the first four tests (2 days) is not counted in the totals because the quantity of permeate produced by the RO cell was not measured during these days. Initially the quantity of renovated wash water used was measured; however, when it was discovered that substantial evaporation losses were occurring, this measurement was abandoned in favor of RO permeate. The average evaporation loss was 16,860/12 or 1,405 grams/man-day.

The average processing rates computed from the tabulated data are:

RO Unit: (99,663 + 42,347)/20.60, or 6,900 gm/hr (15.2 #/hr)

Distillation: 59,127/40.22, or 1,470 gm/hr (3.24 #/hr)

C H E M T R I C

Table 5   <u>PERFORMANCE SUMMARY FOR 70%-YIELD, WASH-LOOP TESTS</u>

Test Day No	RO Permeate Produced	RO Brine Produced	Recycled Flush Water	Input To Still	Flush Water Produced	Recycle Liquor Dumped	Still Purge Losses	Make-Up Water Added	Running Time (hours:minutes) RO	Still
1	---	2520*	1500	3944	3058	0	20	---	3:10	2:30
2	---	3360	1500	4884	3990	330	20	---	2:07	2:09
3	---	2435	1500	3959	3901	109	23	---	1:24	2:48
4	---	3960	1500	5484	4948	84	22	---	2:05	3:07
5	10754	4778	1520	6199	6400	70	(a)	0	2:11	4:30
6	8230	3620	1500	5044	4975	70	42	1760	1:50	3:12
7	8220	3470	1500	4901	4166	72	(a)	2999	1:45	4:05
8	8490	3438	1500	4869	5065	84	58	600	1:45	3:13
9	7185	3129	1500	4560	4469	82	36	2500	1:30	3:12
10	7856	3195	1500	4446	4408	82	36	2000	1:30	2:35.
11	8691	3361	1500	4892	4597	136	35	500	1:45	3:16
12	7090	3285	1500	4566	4568	88	35	2500	1:30	2:51
13	8220	3765	1500	5296	5013	84	47	1000	1:45	3:35
14	9115	3715	1500	4951	4918	76	47	0	1:50	3:20
15	8529	3357	1500	4888	4658	0	(a)	1000	1:45	3:29
16	7283	3234	1500	4515	4143	73	85	3000	1:30	2:55
	99663	42347	18020	59127	57380	917	421	16860	20:36	40:13

*All water quantities are in grams, (a) in next day's total.

Table 6   PERFORMANCE SUMMARY FOR 85% YIELD WASH-LOOP TESTS

Test Day No.	RO Permeate Produced	RO Brine Produced	Recycled Flush Water	Input To Still	Flush Water Produced	Recycle Liquor Dumped	Still Purge Losses	Make-up Water Added*	Running Time (hours:minutes) RO	Still
17	20942**	3702	4500	8135	8063	88	102	0	3:38	5:03
18	18475	3208	4500	7551	7560	86	91	9600	3:10	5:56
19	24515	4293	4500	8786	8348	84	85	2200	4:15	7:00
20	22950	4205	4500	8548	8370	83	73	5500	4:00	6:55
21	26180	4450	4500	8803	8205	82	90	3000	4:30	6:55
22	23165	4040	4500	8383	8230	84	94	6000	4:00	7:09
23	24650	4185	4500	8528	7980	84	95	6000	4:15	6:58
	160877	28083	31500	58734	56756	675	630	32300	27:48	45:56

* To compensate for evaporation losses

** All water quantities are in grams

Table 7 PERFORMANCE SUMMARY FOR 95% YIELD, WASH-LOOP TESTS

Test Day No.	RO Permeate Produced	RO Brine Produced	Recycled Flush Water	Input To Still	Flush Water Produced	Recycle Liquor Dumped	Still Purge Losses	Make-up Water Added	Running Time (hours:minutes) RO	Still
24	27080*	1347	4500	5769	5822	85	73	1700	4:15	5:00
25	25703	1520	4500	6410	5371	436	73	2000	4:00	4:55
26	23867	1265	4500	5668	4220	336	87	5000	3:45	5:02
27	24125	1321	4500	5672	6425	324	95	4000	3:45	4:40
28	23335	1195	4500	5546	5580	250	126	4500	3:40	4:00
29	24865	1243	4500	5597	4230	250	95	3000	3:55	4:00
30	23715	1377	4500	5730	4820	243	66	4000	3:45	3:50
31	20585	1107	4500	5603	5953	292	92	8000	3:20	4:10
32	22970	1248	4500	5534	5210	250	46	3000	3:45	4:05
33	23460	1305	4500	5665	5430	235	78	4000	3:45	3:50
34	25255	1477	4500	5828	5640	307	89	2000	4:00	4:30
35	25145	1712	4500	6065	5215	206	70	3000	4:00	3:50
	290105	16117	54000	69087	63916	3214	990	44200	45:05	51:52

* All water quantities are in grams.

The relatively high distillation rate indicates that too much recycle liquor was dumped - causing the concentration of dissolved solids to be substantially less than 30%. The high recovery rate explains why the purge losses were less than 1%.

In spite of the seemingly excessive dumping of liquor, the mass yield obtained by the distillation unit was 57,380 x 100/ 59,127, or 97%. Thus, even though a solids dryer was not used the water yield was near 98%. However, this exceptional performance was due to the fact that solids were being accumulated in the recycle loop, and these solids displaced some of the water originally contained in this loop. During subsequent tests, when the quantity of solids in the recycle loop reached a quasi-steady value, the water yield obtained by the distillation unit was definitely less than 98%. On the other hand, the overall water yield obtained by the wash loop (RO unit + distillation unit) was always more than 99%.

6.2.1.2 85%-Yield Tests

Table 6 shows that the wash loop was operated for seven days at a 3-man, 85% RO yield condition. Only one test was performed each day because the distillation unit had to process an average of 8,400 grams of waste water (treated flush water + RO brine), which required an average running time of almost eight hours.

The average recovery rate was 56,756/45.93, or 1,235 gm/hr (2.72 #/hr); steady-state rates, of course, were higher because the average includes the start-up and shut-down transients. During this series of tests the daily average recovery rate decreased from 1,470 gm/hr (average for the 70%-yield, wash-loop test) to less than 1,235 gm/hr - as dissolved solids were accumulated in the recycle loop. Higher recovery rates could have been achieved by dumping more liquor, but at the expense of water yield.

The mass yield obtained by the distillation unit was 56,756 x 100/58,734, or 96.6% - instead of 97.0% as obtained during the 70%-yield test. This reduction was anticipated because the concentration of dissolved solids in the 85% yield brine is higher than in 70% brine. Since the mass yield was 96.6%, and less than 675 + 630, or 1305 grams of water were lost in dumping and purging, the water yield must have been in the range of 97 to 98%. However, the overall water yield (for the loop) was still over 99%.

The average processing rate for the RO unit was (160,877 + 28,083)/27.48, or 6,800 gm/hr - which, as expected with a positive-displacement feed pump, is essentially the same as for the 70%-yield test. The evaporation losses were slightly higher

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(32,300/21, or 1,540 g/man-day, versus 1,405 g/man-day), which is probably the result of humidity changes in the test laboratory.

6.2.1.3 95%-Yield Tests

Table 7 shows that the wash loop was operated for twelve days at a 3-man, 95% RO yield condition. During these tests the RO and distillation process times were almost equal - indicating that with an RO yield of 95% the wash loop is capable of supporting up to 18 men, if operated for 24 hours each day.

During this last series of tests the concentration of dissolved solids in the recycle loop reached a critical value - i.e., the recycle flow became too viscous for reliable pumping. Therefore, on day #25 the recycle liquor dump rate was increased to maintain the concentration of dissolved solids at the maximum acceptable level. Consequently, the distillation mass yield obtained was only 63,916 x 100/69,087, or 92.5%. This result is attributed to the fact that the RO brine distilled during this series of tests had a substantially higher concentration of dissoved solids than the brine distilled during the 70% and 85% yield tests. However, the water yield for the entire loop was still over 99%, as evidenced by the fact that the purge losses plus 70% of the dumped mass (target water content) was still less than 1% of the water processed:

Loop Losses = 990 + 0.7 (3214) = 3,240 gm

Loop Input = 290,105 + 16,117 + 54,000 = 360,222 gm

The average evaporation loss during this test was 44,200/12 x 3, or 1,230 gm/man-day - which is slightly less than the losses experienced during the 70% and 85% tests.

6.2.1.4 Permasep Module

This series of wash-loop tests provided an excellent opportunity to evaluate the hydraulic performance of the DuPont Permasep module. Table 8 presents the data necessary for this evaluation, while Figure 18 illustrates the effect of permeation rate and yield on feed pressure - i.e., with a fixed feed rate, the effect of varying brine bleed rate on feed pressure.

Figure 18 shows that for the first 16 man-days of operation (70%-yield tests) the module performed as predicted by the pretest calibration data. However, when the brine bleed rate was decreased to increase the permeation rate for the 85%-yield tests, the resultant feed pressure was 20 to 60 psi higher than predicted by the pre-test calibration data; for the 95%-yield tests, the

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Table 8   HYDRAULIC PERFORMANCE OF PERMASEP MODULE

Test Day No.	Average Yield (%)	Water Permeated (gm)	Running Time (hours)	Permeation Rate (gm/hr)	Feed Pressure (psig) Min.	Feed Pressure (psig) Max.
1	--	--	3.17	--	180	180
2	--	--	2.12	--	---	---
3	--	--	1.40	--	225	240
4	--	--	2.08	--	210	220
5	69.2	10754	2.18	4930	225	235
6	69.4	8230	1.83	4500	220	225
7	70.3	8220	1.75	4700	240	240
8	71.2	8490	1.75	4850	230	235
9	69.7	7185	1.50	4790	230	250
10	71.1	7856	1.50	5240	230	230
11	72.1	8691	1.75	5000	240	250
12	68.3	7090	1.50	4730	210	220
13	68.6	8220	1.75	4700	240	240
14	71.0	9115	1.83	4980	215	240
15	71.8	8529	1.75	4870	260	275
16	69.2	7283	1.50	4860	245	250
17	85.0	20942	3.63	5770	350	380
18	85.2	18475	3.17	5830	335	350
19	85.1	24515	4.25	5770	340	350
20	84.5	22950	4.00	5740	325	345
21	85.5	26180	4.50	5820	340	380
22	85.1	23165	4.00	5790	365	380
23	85.5	24650	4.25	5800	360	380
24	95.3	27080	4.25	6370	470	490
25	94.4	25703	4.00	6430	460	500
26	95.0	23867	3.75	6360	490	520
27	94.8	24125	3.75	6430	495	520
28	95.1	23335	3.67	6360	500	540
29	95.2	24865	3.92	6340	540	560
30	94.5	23715	3.75	6320	550	560
31	94.9	20585	3.33	6220	540	560
32	94.8	22970	3.75	6120	560	575
33	94.7	23460	3.75	6260	520	560
34	94.5	25255	4.00	6310	560	590
35	93.6	25145	4.00	6290	585	610

C   H   E   M   T   R   I   C
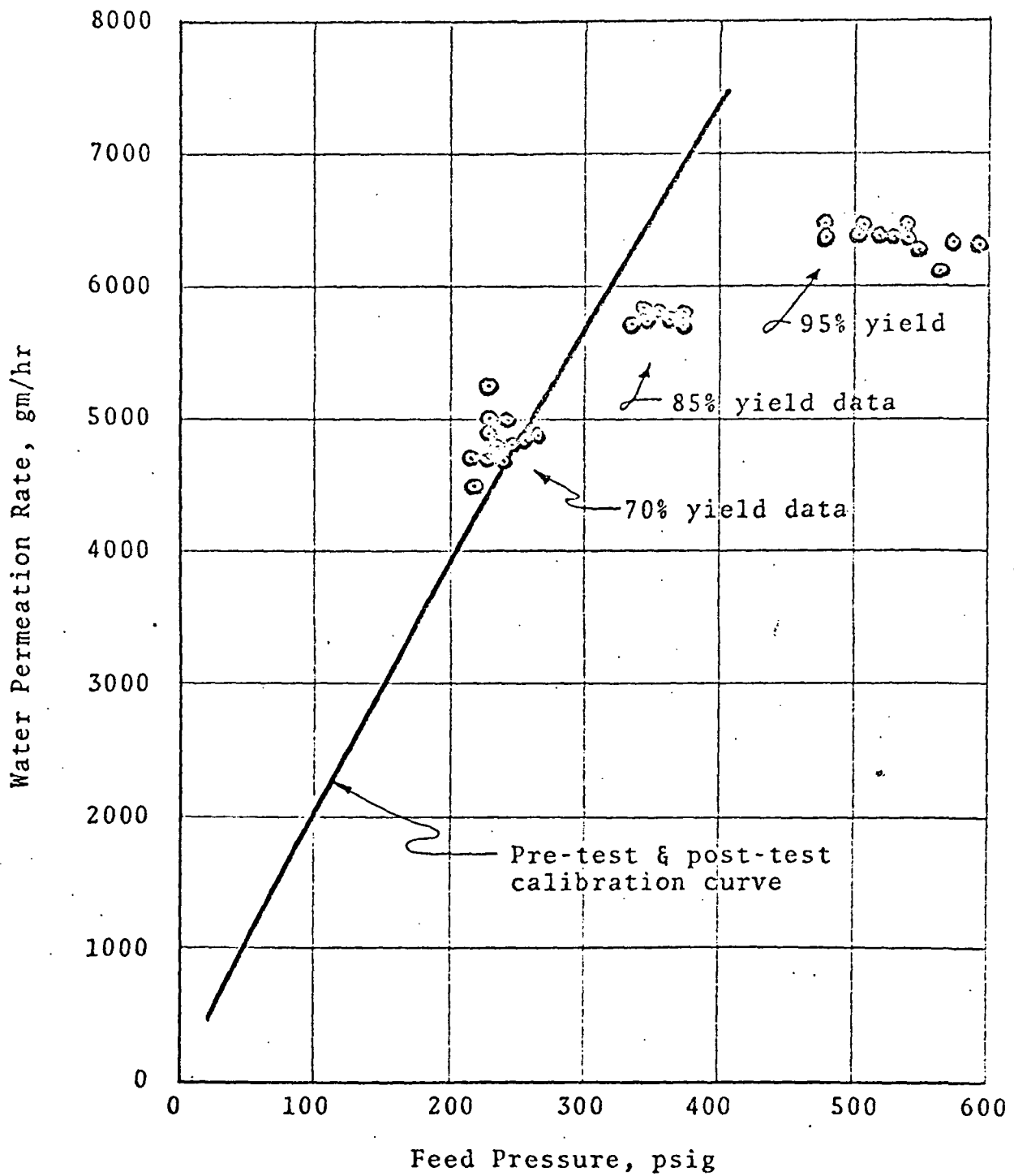
Figure 18   HYDRAULIC PERFORMANCE OF PERMASEP MODULE

C   H   E   M   T   R   I   C

resultant pressure was 120 to 240 psi higher than predicted. In addition, during the 95%-yield test, the resultant cell pressure obviously increased with testing time (see Table 8).

The increasing pressure requirement is attributed to increasing osmotic pressure. This conclusion is indicated by the fact that the brine total solids also increased with testing time (see Figure 19 on page 6-14). if the brine solids had reached a stable concentration, and the feed pressure had continued to increase, the conclusion would had to have been that the RO material degraded or was fouled.

Brine solids concentration alone cannot explain the magnitude of the pressure rise, because the osmotic pressure possible at the measured concentrations is on the order of only 10 psi (the exact value cannot be calculated because the exact composition of the feed water is unknown). The clue to the correct analysis was found during the test when it was determined that the total solids being fed into the module was greater than the amount exiting in the brine and permeate. Since the module was vertically orientated and brine was being withdrawn near the top of the shell, it was then concluded that the brine solids were settling in the shell - like the recycle liquor stratified in the distillation unit's solids filter housing during the previous 30-day test. Therefore, between days 30 and 31 the module was drained, replumbed so that brine was bled from the bottom of the shell, and refilled with used wash water. Subsequent analyses showed that the initial part of the brine drained from the module had a total solids concentration over 18,000 ppm - which substantiated the conclusion that solids were being accumulated in the module as the result of settling, and that the pressure rise was due to increasing osmotic pressure.

Surprisingly, replacing the brine with used wash water, and relocating the brine bleed line did not reduce the resultant feed pressure. Instead, it continued to rise above 550 psi during day 35 - once again indicating that perhaps the module was permanently degraded or fouled. However, an equally surprising result was the fact that brine solids also continued to increase during this test period. From this result it is concluded that the bundle of hollow fibers must not have been adequately flushed of solids when the brine was drained-out at the end of day 30, and that the solids in the bundle continued to exert excessive osmotic pressures. In addition, insoluble matter may have accumulated in the bundle causing radial channeling, and thereby reduced the effective surface area of hollow fibers. In either case, module performance instead of basic material performance was affected. As shown in Section 6.2.4.2, subsequent flushing and recalibration demonstrated that the module had not been permanently degraded. From these results it is concluded that either finer prefilters and/or periodic flushing is required to obtain a reasonably long life for a DuPont Permasep module.

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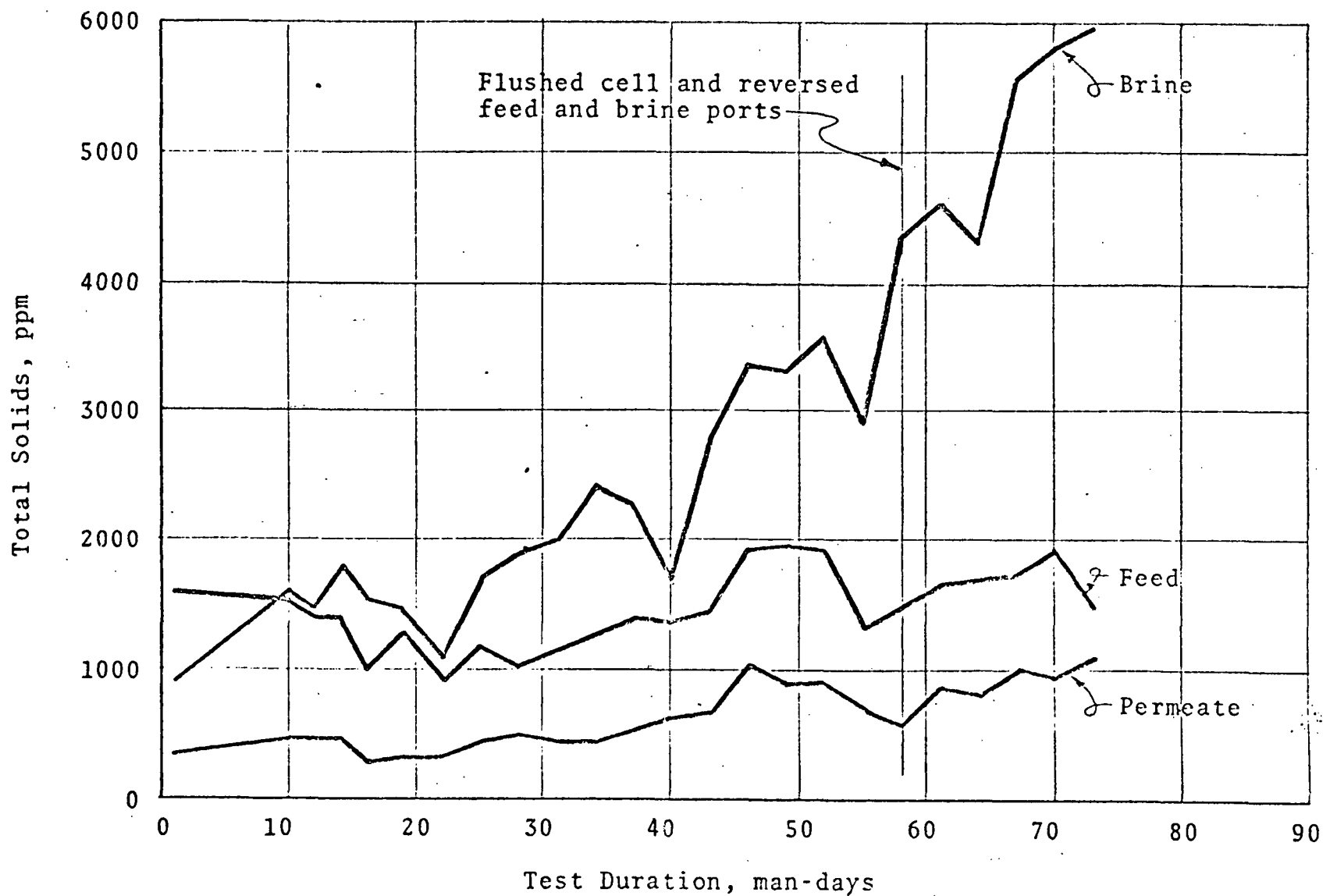
Figure 19   TOTAL SOLIDS IN REVERSE OSMOSIS PROCESS STREAMS

6.2.2 Expendables Required

The expendable items used in the wash loop were:

A. Pretreat Solution (PS)
B. Recycle Filter (RF)
C. Biological Filter (BF)
D. Activated Carbon Filter (ACF)
E. Silver Chloride (AgCl) Columns (2)
F. Liquor Accumulator (LA)
G. Coarse Wash Filter (CWF)
H. Fine Wash Filter (FWF)

Surfactant (Miranol C2M) and germicide (Hyamine 1622) were also expended during this test - but they are not considered to be penalties resulting from the renovation of used wash water.

The PS was added to recycled flush water, to simulate the water demand of a two-stage commode. The quantity of PS added to each 1500 ml batch of flush water ranged from 24 to 31 grams; the average amount was 30 grams/1500 ml, or 2% by weight. Since the commode was unavailable for this test, it cannot be stated that this amount is the exact amount required. This quantity was selected for commonality reasons - i.e., so that the quantity of PS to be stored for treating anal wash water (30 grams/defecation) is the same as the quantity required to treat urine (6 x 5 grams/man-day). During a later test it was determined that the PS required to stabilize urine and avoid deposits in the distillation unit is substantially less than 30 grams/man-day.

A fresh, 25-micron, in-depth filter cartridge was installed in the RF housing at the beginning of this test, and used throughout the test without any evidence of plugging. However, once again the recycle liquor visible within the housing was stratified - indicating that in a weightless state this filter may not achieve as long a useful life as demonstrated during this test.

The BF and the ACF were replaced at the end of days 10, 20 and 30 - as planned. Later in this program the useful biological life of these expendables was increased to at least 30 days.

Two AgCl columns were used; one for saturating filtered condensate with silver chloride ions, and the other for RO permeate. The condensate AgCl column used was the same one used for the 30-day, urine-loop test. The permeate AgCl column was prepared specifically for this test. Since both columns showed no evidence of being exhausted - it is concluded at this point that their useful lives are substantially greater than 193 and 73 man-days, respectively.

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A liquor accumulator (LA), per se, was not used during this test because all of the "dumped" recycle liquor (917 + 675 + 3214, or 4806 grams) was used for total solids determination. The average specific gravity of this liquor was near 1.15. Therefore, if all of the dumped liquor had been accumulated, as would be necessary on a spacecraft without overboard dumping, the required LA volume would have been 4806/1.15, or 4190 ml (255 in^3). However, this computation does not account for the solids accumulated in the recycle loop (e.g., filter housings). A more accurate computation for the required LA volume on long duration missions can be made by using the 95%-yield test data - i.e., the dumped volume per man-day after the concentration of dissolved solids in the recycle loop reached the maximum level acceptable for pumping recycle liquor. This number equals 3214/36 x 1.15, or 77.5 ml/man-day (4.72 in^3/man-day). If the RO unit does not retain part of the wash water solids the LA specific volume will be greater than 77.5 ml/man-day.

The wash-loop test was initiated using two Ronningen-Petter, back-flushable, surface filters - one with a 700 mesh (30 micron) stainless steel screen, and the other one with a 1 - 3 micron polypropylene screen over a stainless steel supporting screen. On day 14, both of these filters plugged, as indicated by pressure gages on both sides of each filter. The filter elements were then removed, cleaned with a brush, and reinstalled. On day 15, they plugged again - indicating that the interstitial spaces could not be cleaned by scrubbing. These filters were then back-flushed, using the RO feed pump. However, this did not solve the problem, so at the end of day 16 they were replaced with two in-depth filters - namely, Cuno 10 and 3 micron Micro-Wynd cartridges in Model 1M housings. The in-depth filters performed satisfactorily for the remainder of the test - however, the pressure drop across each filter increased from less than 1.5 psi on day 17 to approximately 4 psi by the end of day 35. Therefore, it is concluded that the useful life of these filters is only slightly more than 7 x 3 + 12 x 3, or 47 man-days.

6.2.3 Water Quality

The analytical work performed during this test was purposely concentrated upon determining (1) the total solids, and (2) the inorganic and organic content of the RO process streams. Spot checks were made of the critical parameters for the distillate.

6.2.3.1 RO Process Streams

Grab samples of the feed, brine and perm-
eate streams were taken each day for total solids determinations;
the results of these measurements are presented in Figure 19.
As shown, and discussed in Section 6.2.1.4, the brine solids in-
creased with test duration - even though the Permasep module was
drained at the end of the 30th day. The explanations for this,
of course, are that the bundle of hollow fibers serves as in-
depth filter and/or is susceptible to radial channeling. Con-
sequently, the permeate solids also increased with test dura-
tion - so that after 35 man-days they exceeded the 500 ppm limit
recommended by the U. S. Public Health Service and specified by
NASA MSC Specification C35 for potable water. It is not known if
renovated wash water must be potable - but if it must be, this
test has demonstrated that the permeate will have to be charcoal
filtered. Other alternatives are to use a different RO module
and/or a germicide which is rejected more easily than Hyamine
1622; finer prefilters may also improve the quality of the perm-
eate.

Analyses for the organic and inorganic content of the RO
processes streams was initiated on the 24th day - when, on the
basis of total solids, it appeared as though the feed stream
composition had attained equilibrium. The results of the anal-
yses performed at the NASA MSC on a Beckman Carbonaceous Analyz-
er are presented in Table 9. Inspection of the values listed
reveals a gradual increase with test duration - and that only
about 40% of carbonaceous matter fed into the module was reject-
ed. However, the data also reveals the accumulation of carbona-
ceous matter within the module - i.e., the carbon content of the
brine is only two to three times greater than for the feed, when
it should be almost nine times greater. Thus, the carbon analy-
ses substantiates the bundle accumulation hypothesis drawn from
the total solids analyses.

The poor rejection performance of the Permasep module is
partly due to the use of Hyamine 1622. Table A-6 (on page A-16)
shows that DuPont's Permasep material rejects only 39.9% of a
similar germicide (Hyamine 10-X). Thus, if a Permasep module is
required to produce "potable" wash water (COD $<$ 100 ppm, as rec-
ommended by the Ad Hoc Committee), a more rejectable germicide
and/or charcoal filtration will be required.

A sample of permeate from day 35 (calendar day #27) was
submitted to the MSC for analyses similar to that performed for
potable water in the urine-loop test. The results of the metal
analyses indicate the presence of only one metallic species -
namely, zinc, and at very low concentration (0.3 ppm). The sil-
ver content was less than 0.05 ppm; it should have been close to
1.0 ppm (the silver dose provided by the AgCl column). The perm-

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Table 9   CARBON CONTENT OF RO PROCESS STREAMS
(95%-Yield Tests)

Test Day No.	Total Carbon*			Organic Carbon			Inorganic Carbon		
	F.Feed**	Brine	Perm.	F.Feed	Brine	Perm.	F.Feed	Brine	Perm.
24	1016	2258	568	980	2200	550	36	58	18
25	1023	--	662	990	--	620	33	--	42
26	1338	2265	630	1300	2200	610	38	65	20
27	1337	2468	730	1300	2400	710	37	68	20
28	1238	2771	781	1200	2700	760	38	71	21
29	1232	2679	762	1200	2600	740	32	79	22
30	1236	3374	784	1200	3300	760	36	74	24
31	1342	2485	682	1300	2400	660	42	85	22
32	1242	2981	726	1200	2900	700	42	81	26
33	1440	3682	742	1400	3600	720	40	82	22
34	1340	3483	822	1300	3400	800	40	83	22

* All values are ppm

** Filtered Feed

eate contained 200 ppm chloride which is sufficiently high to cause a low silver dose via the common ion effect. Aside from the carbonaceous contaminants (TOC = 745 ppm) the major contaminant detected was chloride, although 15 ppm sulfate was also found. Nitrate and nitrites were absent, but 3 ppm ammonia was present. The measured anion content would suggest a 200 to 350 ppm sodium/potassium content - although the possibility exists that all or part of the cationic load could be organic cations (Hyamine 1622 is a cationic germicide). The pH and specific resistance values were 7.79 and 5,000 ohms, respectively.

Sterility tests were conducted on all streams on 20 of the 27 calendar days involved. The feed stream in every test showed very heavy turbidity in less than 24 hours and in some cases, less than 16 hours - thereby indicating very substantial microorganism densities. The brine was sterile on two test days and non-sterile on the remaining days tested; the slow development and low degree of turbidity noted in most tubes indicate a bacteria density substantially lower than the feed density. The silver-dosed permeate was sterile on 16 of the 20 days tested. On four of the test days, a sparse turbidity developed nearly immediately and remained essentially unchanged throughout the 48-hour incubation period; the immediate onset of turbidity suggests a sample-media incompatability; however, biological contamination cannot be ruled out.

A total of twelve men used renovated wash water and Miranol C2M for showering. All of them agreed that only about one-half of the water available was necessary, and that they were adequately cleaned. Even though the renovated water was "sudsy", most of them "felt" that they were too clean - as evidenced by a dry-skin feeling. Finally, on the 26th day, one of the users developed skin flaking and peeling in areas which are difficult to flush (rinse); eventually, two more users developed the same problem. Only one of the three men affected was a frequent user of the shower - while two other frequent users did not acquire the problem. Also, when the affected men stopped using the shower, the dermatological problem dissappeared within five days.

6.2.3.2 Distillate

Extensive chemical analyses were not performed on the raw condensate nor the flush water (filtered condensate) because of the analytical effort required to support the RO tests. However, the raw condensate was tested for sterility on 22 of the 30 test days. In each of the 22 tests, all five thioglycolate tubes failed to show turbidity even after 72 hours incubation, thereby indicating sterility. Spot checks of raw condensate and flush water were made with pH and specific resistance measurements. The raw condensate was characterized by a pH of 4.5 to 5.5 and specific resistances of 20 to 40 Kohms-cm.

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The raw condensate on most days had a distinct, and in some cases a very strong, iodine odor. This iodine odor is due to co-distillation of iodine from the recycle liquor which lacked sufficient organic contaminant. As a result, the iodine concentration remained sufficiently high to favor high iodine partial pressures in the evaporator. Normally, the impurities in the flush water would have reduced iodine (as I_2) to iodide ion ($2I^-$) leaving only a small, but biologically effective, iodine concentration; in the presence of excess iodide, iodine combines with the iodide to form the triiodide ion (I_3^-).

The flush water was characterized by pH values near 4 and specific resistance near 20 Kohm-cm. The flush water was sterile on each of the 22 days tested.

6.2.4 Recalibration Tests

The distillation unit and the RO module were both recalibrated on days 28, 29 and 30 (Tests 36, 37 and 38). After flushing, the performance of both units returned to the pre-test level, as indicated by the following summaries.

6.2.4.1 Distillation Unit

The three recalibration tests were performed using tap water, and dumping the excess feed to flush the evaporator and avoid recycling concentrated tap water. The running time ranged from 4:35 to 5:25 hours. By varying the cabinet temperatures, the following performance was achieved after the condenser pressure reached a fairly consistent level.

Day No.	Condenser Pressure	Recovery Rate	Input Power
28	33-37 torr	1301 g/hr	96-102 watts
29	35-39	1402	102-108
30	37-40	1603	103-107

6.2.4.2 RO Module

Deionized tap water saturated with silver chloride ions was used to recalibrate the Permasep unit. The first test was performed over a one hour period. During the second and third days the feed and bleed rates were both held constant for periods of approximately three hours. The results obtained are summarized as follows.

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Day No.	Bleed Rate	Permeation Rate	Feed Pressure
28	2880 g/hr	3300 g/hr	275 psig
29	1400	5170	390
30	960	6100	385

Comparison of these data to the calibration curve presented in Figure 14 (on page 4-6) indicates that during the 28th day the resultant feed pressure was 70 to 100 psi higher than calibrated level; essentially the same result was obtained during the 29th day. However, on the 30th day the permeation rate increased - indicating that the bundle was finally being cleaned-out, and that the hollow fibers had not been degraded by renovating 73 man-days of used wash water. When the cell was calibrated again for the test described in Section 9 of this report, the performance was exactly as predicted by Figure 14.

6.3 Conclusions & Recommendations

The 30-day, wash-loop test achieved it's primary objective - namely, "to demonstrate reliable operation of a reverse osmosis subsystem processing used wash water, and feeding brine to a compression distillation subsystem - which also receives treated anal wash water from a two-stage fecal collector". The fecal collector had to be simulated - but since the flush water was heavily dosed with pretreatment solution which is more difficult to process when the anal wash water does not contain any other dissolved solids, the test is considered to be valid. Secondary objectives which were also achieved include:

1. Demonstrating that when Miranol C2M and Hyamine 1622 are used as surfactant and germicide, respectively, used wash water renovated by DuPont B-5 Permasep module can have more than 1000 ppm total solids and 800 ppm total carbon.

2. Demonstrating that Miranol C2M is an effective surfactant - however, it can cause dermatological problems in areas which are difficult to flush or rinse.

3. Demonstrating that if Hyamine 1622 is added to clothes wash and dish wash water, at a 5000 ppm concentration, and these waters are mixed with used shower water, the resultant mixture is not sterile. However, the permeate produced by a DuPont B-5 Permasep module processing this mixture can be sterilized by dosing with AgCl ions.

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New design data obtained during this test include:

1. The recycle-to-condensate flow ratio required to distill concentrated wash water is higher than for concentrated urine. Apparently, wash water solids have a greater effect on viscosity than urine solids.

2. The rollers required in a peristaltic-type of feed pump should be gear-driven to avoid abrasive wear of the pump tubing.

3. The average evaporation loss when showering and washing dishes and clothes in CHEMTRIC's laboratory is in the range of 1230 to 1504 g/man-day.

4. The water yield obtainable with the wash loop is greater than 99% - even though purge vapor is lost and recycle liquor is dumped to an accummulator.

5. The liquor accumulator volume required to dispose of wash water solids is 77.5 ml/man-day.

6. Surface-type prefilters are ineffective; if a 10 and 3 micron in-depth filters are used in series they have a useful life greater than 47 man-days.

7. DuPont's B-5 Permasep module tends to accumulate solids, which affect the permeation rate; therefore, the feed must be filtered to less than 3 microns, or the module periodically flushed to the distillation unit to obtain a useful life greater than 73 man-days.

From the results of this test it was concluded that more work is required on reverse osmosis before it can be positively stated that this process is acceptable for renovating wash water onboard manned spacecraft. DuPont's B-5 Permasep material still appears to be the most compatible RO material - but in a fiber-bundle configuration with radial flow it is easily "fouled" with solids and/or gels capable of passing through a 3 micron filter. In addition, when 5000 ppm of Hyamine 1622 is used as the germicide in clothes and dish water which is mixed with used shower water - the resultant mixture is non-sterile, and the permeate contains approximately 1000 ppm of solids and carbon. Other, more easily rejected germicides should be investigated. Based on these recommendations, Contract NAS9-9191 was ammended to include the additional wash loop tests reported in Section 9.

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PRETREATMENT REDUCTION TESTS

The quantity of pretreatment solution (PS) used during the 30-day urine-loop and wash-loop tests ranged from 1.8 to 2.0% of the urine and anal wash water treated. Based upon previous experience it was known that by using this quantity the waste waters would be sterile, and that the surface of the evaporator in the distillation unit would remain clean. Lessor quantities were not investigated because the primary purpose of these tests was to demonstrate reliability.

The daily quantity of urine and anal wash water anticipated for each man is 6 x 250 + 1500, or 3000 grams. Therefore, on a modular space station which is resupplied every 180 days the storage volume required for pretreatment solution, to be used at a 2% rate, would be 3000 x 0.02 x 180, or 10,800 ml (660 in^3)/man. Obviously, this becomes a significantly-sized expendable on multi-manned space stations. Since it was believed that dosing rates less than 2% would be satisfactory, contract NAS 9-9191 was amended to include the work necessary to determine the minimum quantity of PS required. The following subsections show that the minimum quantity actually required is only 0.8% of the waste water to be treated - as demonstrated by static tests with urine, and another 30-day test of the urine-loop.

The secondary objective of this series of tests was to extend the useful life of the activated carbon filter used to post-treat distillate. As shown in Section 7.3, this was accomplished. In addition, it was discovered that prepasteurizing the deionizer avoided the biological contamination problem encountered during the first 30-day, urine-loop test.

7.1 Background

The pretreatment solution (PS) is based on a previously de-veloped formulation which contained (1) Wescodyne concentrate, (2) sulfuric acid, (3) silicone antifoam, and (4) water. Each of the above ingredients, except water, participates in treating urine; water is necessary in the formulation to moderate acidity and to reduce the viscosity of the formulation.

Wescodyne is an iodophor-type disinfectant which contains 10% available iodine. This germicide utilizes the broad-spectrum, rapid-kill capabilities of iodine. Iodine typically is lethal to yeasts, fungi, bacteria, protozoa, rickettsia and virus in 10 to 100 ppm concentrations. In standard tests of bactericidal effi-ciency, 10 ppm iodine typically kills 99.9999% of bacteria such as Escherichia coli and Micrococcus pyogenes in 30 seconds or less. The only known disadvantage of iodine is that the effective species, namely the diatomic molecular form (I_2) and the hypoio-dous acid (HIO), are subject to chemical reduction by even extreme-ly weak reducing agents. In addition, I_2 will hydrolyze under

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alkaline conditions producing HIO; exposure to moderate light causes decomposition of the hypoiodous acid to iodide. The above problems can be counteracted by (1) adding sufficient excess of iodine over and above that amount consumed by the reducing agents in the treated medium, and (2) blending the iodine with an acid.

The function of the sulfuric acid in the formulation is not only to prevent iodine hydrolysis but also to "fix" ammonia in the treated medium. Sufficient acid must be added via the PS to shift the waste pH to a value less than 4; at or below this level, hydrolysis is retarded and the ammonia partial pressure becomes negligible.

The antifoam serves to control the foam head height of normal urine and waste water, and thereby minimize the possibility of carry-over in the distillation unit. On the other hand, the iodophor tends to increase both foam head height and foam stability. Therefore, sufficient antifoam must be added to counteract the natural foaming capability of urine as augmented by the iodophor.

The concentration of each ingredient in the formulation, as well as the formulation dose level for urine, was previously established by experiment under Contract AF33(615)-2124; the composition selected is as follows:

Iodophor	43.5%
Sulfuric Acid	6.1%
Antifoam	2.4%
Water	48.0%

The above values are based on anhydrous acid, anhydrous antifoam, and the as-received iodophor. The dose level was established at 20 grams of PS per liter of urine; this dose level resulted in a 14 ppm iodine residual at the end of a 14-day storage period during which no viable bacteria were detected.

Tests conducted under the Air Force contract revealed an additional benefit and an additional disadvantage. On the plus side, the formulation prevented fouling of the evaporator and appeared to retard the generation of precipitated solids. On the negative side, the iodophor contained a volatile organic substance which was eventually identified as an alcohol. The manufacturer (West Chemical Laboratories) revealed that the iodophor contained 5% - 10% isopropanol which was added to reduce viscosity. In prior consultation, the manufacturer had indicated that Wescodyne contained only iodophor and inert ingredients.

For the NAS 9-9191 program, an alternate iodophor was selected - namely, Biopal VRO-20 (General Aniline & Film). Discussions with the supplier revealed that Biopal is the active ingredient in Wescodyne, and that Biopal does not contain any volatile organics. The product is defined as a nonylphenoxypolyethylenoxy-

iodine complex with trace quantities of water as the major impurity. The product ~~~~ d ~~ a minimum of 20% available iodine, which is twice the ~~~~ ~~ oncentration in Wescodyne; the organic portion of the com~~~~ ~~~ surfactant similar to an ingredient used in many indus ~~~~ ~~ength cleaners.

Substituting Biopal VRO-20 into the formulation did not present a major chemical problem. However, to insure success, the new formulation had to provide both the desired surfactant and germicidal properties. This requirement produced the following dilema: If the new formulation provided a Biopal quantity equal to the Wescodyne quantity, then a formulation with twice the required iodine quantity would result; if the new formulation was blended to provide an identical iodine concentration, then only 1/2 of the previous surfactant quantity would be provided. It was decided to duplicate the formulation on the basis of iodophor content rather than iodine, for the following reasons:

1. The surfactant portion of the iodophor undoubtedly plays a key role in preventing evaporator fouling; therefore, demonstration of reliable operation would be more assured by maintaining a proven ratio of surfactant-to-dissolved solids.

2. The doubled iodine concentration provided a substantial contingency margin - which should not be detremental to performance of the distillation unit.

The quantity of iodophor, acid and antifoam used under Contract AF33(615)-2124, for treating the urine collected during each micturation, was:

Iodophor	2.45 gms
Sulfuric Acid	0.34
Antifoam	0.13

The quantity of water used in the formulation was 2.69 grams, which corresponds to 48% of the solution mass. For this program it was decided to also investigate the effect of water concentration on the preparation and storability of the solution, and determine the minimum concentration required. It was found that at water concentrations above 55% the solution tends to stratify - while at concentrations below 40% it is too viscous. Therefore, it was decided to fix the water concentration at 41.6%, so that the solution mass required per micturation would be exactly 5.0 grams.

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Ingredient	%	Mass/Micturation
Biopal VRO-20	49.0	2.45 grams
Sulfuric Acid	6.8	0.34
Antifoam	2.6	0.13
Water	41.6	2.08
	100.0	5.00 grams

The following subsections describe the static and dynamic tests performed to determine if less than 5.0 grams of PS can be used per micturation to (1) stabilize urine for storage at room temperature, and (2) avoid fouling the evaporator surface in the distillation unit.

7.2 Static Tests

Urine collected onboard a modular space station will undoubtedly be transferred to a waste storage tank, from where it will be transferred on demand to the water recovery unit. With this arrangement fresh urine is mixed with older urine - and the contents of the storage tank may have to be held at cabin temperature for many days during an emergency situation. To avoid gross biological activity and subsequent failure of the water recovery equipment, all of the urine must be dosed with the selected pretreatment solution while it is being collected and transferred to the waste storage tank. The objective of the tests described hereafter was to determine the minimum quantity of iodophor (Biopal VRO-20) needed in the PS to meet this requirement.

The average quantity of urine anticipated during each micturation on a modular space station, as specified by Contract NAS 9-9191, is 260 grams. However, the maximum quantity may be as large as 520 grams - and treated urine may be diluted with humidity condensate in the waste storage tank. Therefore, the static tests were performed with two urine volumes - namely, 260 and 520 grams. A raw urine pool was accummulated in a refrigerator over a 24-hour period so that the urine aliquots used in the tests would be identical. Each urine volume was placed in a sterilized bottle containing 150 ml of deionized tap water saturated with silver chloride ions (simulated flush water). PS containing 1/2, 1/4, 1/8 or 1/16 of the Biopal quantity (2.45 gm) used during the first 30-day urine-loop test was added to each bottle. The quantity of sulfuric acid and antifoam added to each bottle was the same as before (i.e., 0.34 and 0.13 grams) - because the relatively small amounts required have a negligible effect on storability and expendable weight penalty. The bottles were stored on a lab bench at room temperatures - and inspected daily for gross changes in color and/ or turbidity over a 10-day period. Aerobic plate counts (pour plate method using trypticase-soy agar) were performed when visual inspection indicated urine decomposition, or at the end of the 10-day storage period; the 10-day period was selected arbitrarily as a reasonable duration for putrifaction to develop.

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The results of the static tests are tabulated in Table 10.
As shown, the urine dosed with 1/2 and 1/4 the previous quantity
of Biopal were sterile at the end of the 10-day test period; at
lower dosages growth was observable within five days. The 1/4
dose level appears to be marginal because the 520 gram sample was
slightly cloudy after 10 days. From this it could be concluded
that a dose greater than 1/4 should be used to provide a safety
factor. However, since the 520 gram sample is considered to be
double the average quantity, a 1/4 dose provides a safety factor
of nearly 2 when the average urine quantity is 260 gm/micturation.

Table 10 RESULTS OF VARIABLE PRETREAT STATIC TESTS

Biopal Dose (ratio)	Urine Mass (gm)	Aerobic Count (No/100 ml)	Odor	Appearance
1/2	260	0	Normal	Clear
1/2	520	0	Normal	Clear
1/4	260	0	Normal	Clear
1/4	520	0	Normal	Slt. Cloudy
1/8	260	32	Putrid	Heavy Sediment
1/8	520	3.9×10^5	Putrid	Heavy Sediment
1/16	260	2.0×10^6	Putrid	Heavy Sediment
1/16	520	1.5×10^6	Putrid	Heavy Sediment

From the results of the static tests it was concluded that
dynamic tests should be performed to evaluate both the 1/2 and
1/4 dose levels. The recommended PS formulations were as follows.

Ingredient	1/2 Dose	1/4 Dose
Biopal VRO-20	1.23 grams	0.61 grams
Sulfuric Acid	0.34	0.34
Antifoam	0.13	0.13
Water	1.30	1.17
	3.00 grams	2.25 grams

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7.3 Urine-Loop Test

Dynamic evaluation of reduced PS concentrations was necessary because one of the functions of the iodophor in the formulation is to prevent fouling (scaling) of the evaporator surface. Therefore, another 30-day, urine-loop test was performed to achieve this objective. Secondary objectives were to (1) prolong the useful life of the activated carbon filter, and (2) evaluate the use of a deionizer which can be pasteurized. All of these objectives were achieved, as evidenced by the following summary.

7.3.1 Test Set-Up & Procedure

This test was performed using the equipment and arrangement illustrated in Figure 20. This is essentially the same set-up as described in Section 5 for the first 30-day, urine-loop test; the only change made was removal of the solids dryer and it's supporting equipment.

As in the previous 30-day, urine-loop test, 24 one-liter bottles containing 150 ml of flush water and the prescribed amount of PS were prepared each day for collecting urine. Initially, the 1/2 dose (3.0 grams of PS) was used. When this concentration indicated no operating or water quality problems after 7 days of operation, the dose level was reduced to 1/4 (2.25 grams of PS). When this proved to be acceptable, no PS was used for two days, followed by another 3 days at the 1/4 iodophor level - to determine if this level was adequate to resterilize the recycle loop after it was purposely contaminated. Finally, the loop was run for 11 more days at the 1/4 level to complete 30 days of operation at the 4-man process rate. The exact PS formulations used are presented on page 7-5.

Initially, the bacteria filter (BF) and the activated carbon filter (ACF) used for post-treatment were the same as the ones used during the previous tests. At the end of the 14th day they were both replaced; however, the new BF was packed with a mixture of AgCl particles and glass beads - to determine if saturating the raw condensate with AgCl ions would extend the useful life of the BF and the ACF.

The deionizer provided to convert flush water to potable water was specially prepared for this test. After the resins were washed the deionizer was assembled, connected to a filter-sterilized deionized water source, and immersed in a 180°F water bath. A low water flow rate was maintained continuously; the deionized water was pre-heated to bath temperature prior to entering the deionizer by passage through a 12-foot length of type 304 stainless steel tubing lying in the water bath. The deionizer was subjected to these conditions for 18 hours prior to installation in the post-treatment loop.

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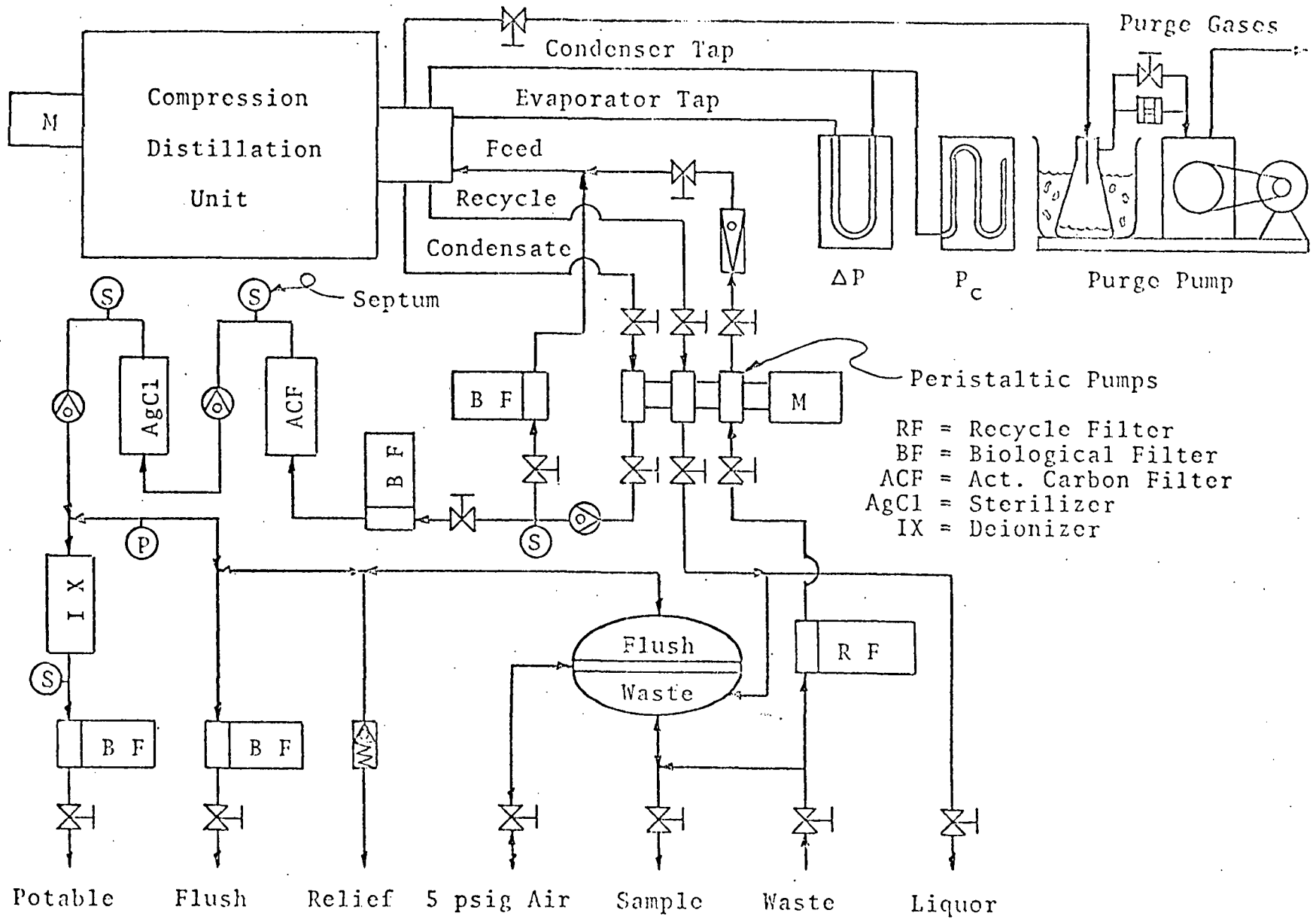
Figure 20   FLOW SCHEMATIC FOR PRETREATMENT REDUCTION TEST

Recycle liquor was dumped each day whenever a "running" calculation indicated that some should be withdrawn to avoid total solids concentrations above 30%. The dumped liquor was used to measure the actual concentration of total solids - and adjust the running calculation and the quantity dumped each day.

Routine chemical analyses were performed in-house on raw condensate, ACF effluent and potable water. Samples of each of the above were also shipped to the NASA MSC for more extensive analysis.

Major emphasis was placed on microbiological analyses. Such analyses were routinely performed almost immediately. The membrane filter technique was employed for making all microbiological counts with the exception of anaerobic counts. Anaerobic counts were determined on the basis of a multiple tube dilution method, employing thioglycolate broth and 56 ml of inoculum. Standard Methods Agar, endo agar, and rose bengal-malt extract agar were used as the nutrient medium for aerobic, coliform and yeast mold counts, respectively. Sterility tests, employing 10 to 20 ml of inoculum in thioglycolate broth, were routinely done to back up aerobic counts.

7.3.2   Results & Discussion

The urine loop was operated at the four-man processing rate for 30 days, plus one day for recalibration. The qauntity of PS was varied according to the following schedule.

```
Day  1 through  7 - 3.0 grams/micturation
     8 through 14 - 2.25    "         "
    15 through 16 - 0.00    "         "
    17 through 19 - 2.25    "         "
    20 through 30 - 2.25    "         "
```

At the end of the 7th, 14th and 19th days the waste tank, distillation unit and recycle loop were flushed with deionized water, and the recycle filter replaced, to remove residual iodine and urine solids prior to starting the next test sequence. Prior to starting the 20th day the distillation unit had to be partially disassembled to remove urine solids which had accummulated during the 15th and 16th days when PS was purposely omitted from the collection bottles to determine the effect of untreated urine on thermodynamic performance and water quality. On the 23rd day the roll pin in the compressor drive coupling broke and had to be replaced; this failure is attributed to improper reassembly on the 20th day. No other problems were encountered, except for the usual need to replace the tubing in the peristaltic pumps about every 15 days (60 man-days).

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A three-hour r̶e̶ ̶d̶i̶s̶t̶i̶l̶l̶a̶t̶i̶o̶n run with deionized water, after the 30th day, indicated that the thermodynamic performance of the distillation unit was the same as before the first 30-day, urine-loop test.

7.3.2.1 Thermodynamic Performance

Table 11 presents the data obtained to evaluate the effect of PS concentration on the thermodynamic performance of the distillation unit. The four test sequences performed are subtotaled because the waste tank, distillation unit and recycle loop were flushed after each sequence. The anamolies and trends evident in this data are explained as follows.

1. During the first three test days the quasi-steady state recovery rates were exceptionally high. This is attributed to the fact that the distillation unit had been disassembled for inspection after the 30-day, wash-loop test, and the compressor was regreased prior to reassembly. Thus, the internal clearances were reduced below their normal running values, and approximately 20 hours of running time (3 test days) was necessary to obtain normal performance. This result, however, indicates that substantially higher performance is obtainable by reducing internal clearances in the compressor.

2. The purge losses required to maximize recovery rate when processing untreated urine (days 15 and 16) are substantially higher than the average of the other days. Thus, the pretreatment chemicals obviously reduce the generation of non-condensable gases produced by the putrification and/or fermentation, which occurs with untreated urine.

The yields obtained during these tests are summarized as follows.

Days	Dose	Mass Yield	Water Yield
1 - 7	1/2	92.0%	95.7
8 - 14	1/4	93.5	95.7
20 - 30	1/4	95.6	96.6

Theoretically, lower dose levels should permit the attainment of higher yields - because less recycle liquor would have to be dumped to limit the concentration of dissolved solids to the level necessary to achieve a specified minimum recovery rate. The table above indicates that the yield obtained during the 1/4-dose level test was equal to or greater than the yields obtained for the 1/2 dose level tests. However, Table 11 indicates that the recycle liquor dumped during days 23 through 30 contained

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Table 11   PERFORMANCE SUMMARY FOR PS REDUCTION TEST

Test Day No.	Biopal Dose Level	Waste Input (grams)	Water Output (grams)	Purge Loss (grams)	Liquor Dumped (grams) Water	Solids	Recov. Rate (kg/hr)	Specific Energy (w-h/kg)
1	1/2	9173	8778	80	0	0	1.78	55
2	"	8887	8155	78	0	0	1.66	57
3	"	8012	7540	67	0	0	1.62	64
4	"	7871	7390	80	260	183	1.20	88
5	"	8068	7205	64	538	228	1.46	66
6	"	8148	7283	56	536	229	1.42	68
7	"	8026	7194	63	576	164	1.42	68
Sub-Totals		58185	53545	488	1910	804	--	--
8	1/4	9426	9204	79	0	0	1.44	64
9	"	9559	9099	94	0	0	1.53	59
10	"	8825	8540	91	0	0	1.47	66
11	"	8883	8180	92	507	160	1.52	59
12	"	9063	8295	99	488	192	1.34	70
13	"	9059	8229	119	495	188	1.18	75
14	"	10038	9077	90	585	178	1.40	66
Sub-Totals		64853	60624	664	2075	718	--	--

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(Table 11 Continued)

Test Day No.	Biopal Dose Level	Waste Input (grams)	Water Output (grams)	Purge Loss (grams)	Liquor Dumped (grams) Water	Solids	Recov. Rate (kg/hr)	Specific Energy (w-h/kg)
15	None	10100	10041	160	0	0	1.34	68
16	"	11182	11162	175	0	0	1.29	70
17	1/4	9644	9455	75	0	0	1.37	64
18	"	8533	7957	82	0	0	1.30	70
19	"	9551	8885	149	0	0	1.24	79
20	1/4	10054	9501	171	0	0	1.39	68
21	"	10501	9900	130	0	0	1.30	68
22	"	9456	8253	121	0	0	1.39	66
23	"	9459	8636	120	477	174	1.19	77
24	"	9145	8688	115	277	83	1.44	64
25	"	9740	9884	111	176	64	1.42	66
26	"	9978	9444	116	0	0	1.29	75
27	"	9221	8745	99	170	99	1.30	79
28	"	9186	8020	110	356	183	1.19	81
29	"	9663	9726	125	221	146	1.14	90
30	"	10503	9636	103	482	421	1.06	84
Sub-Totals		106906	100433	1321	2159	1170	--	--

more solids tha⋯⋯ or dumped during the other tests. Thus, the higher yiel⋯⋯ ⋯hieved during the last test series because of insuffi⋯⋯ ⋯umping - instead of the theoretical need for less dumping. From these results it must be concluded that in the range of dose levels of interest, the experimental data does not positively indicate a significant advantage for the 1/4-dose level over the 1/2-dose level.

This series of tests provided the opportunity to experimentally determine the effect of pretreat and urine solids concentration on recovery rate - because all of the recycle liquor withdrawn (dumped) from the recycle loop was available for total solids determinations. Figure 21 presents the results obtained, in graphical form, to illustrate (1) how recovery rate is depressed by total solids in the recycle liquor, and (2) that PS dose level has no apparent effect on recovery rate at a given total solids concentration.

The relatively large deviation of the test data presented in Figure 21 is due to the difficulty in obtaining a representative sample of the recycle liquor. Since the total volume of the recycle loop was relatively small (about 10 liters), and treated urine and flush water were periodically added to this loop - the time of sample withdrawal (dump) had a significant effect on this concentration of total solids. Fortunately, the data is accurate enough to establish the trend, and indicates that PS dose level does not have a significant effect. Later in this program the volume of the recycle loop was increased to obtain more accurate data on the effect of solids on recovery rate (see Section 8).

7.3.2.2 Expendables Required

The expendable items used in this urine-loop test were:

A. Pretreatment Solution (PS)
B. Recycle Filter (RF)
C. Biological Filters (BF)
D. Activated Carbon Filter (ACF)
E. Silver Chloride (AgCl)
F. Deionizer (I-X)
G. Liquor Accummulator (LA)

The quantity of PS used over the 30-day period was varied, of course, to determine the effect of PS dose level on thermodynamic performance and water quality. For the 1/2 and 1/4 dose level tests the quantity of PS added to each urine collection (micturation) bottle was 3.00 and 2.25 grams, respectively.

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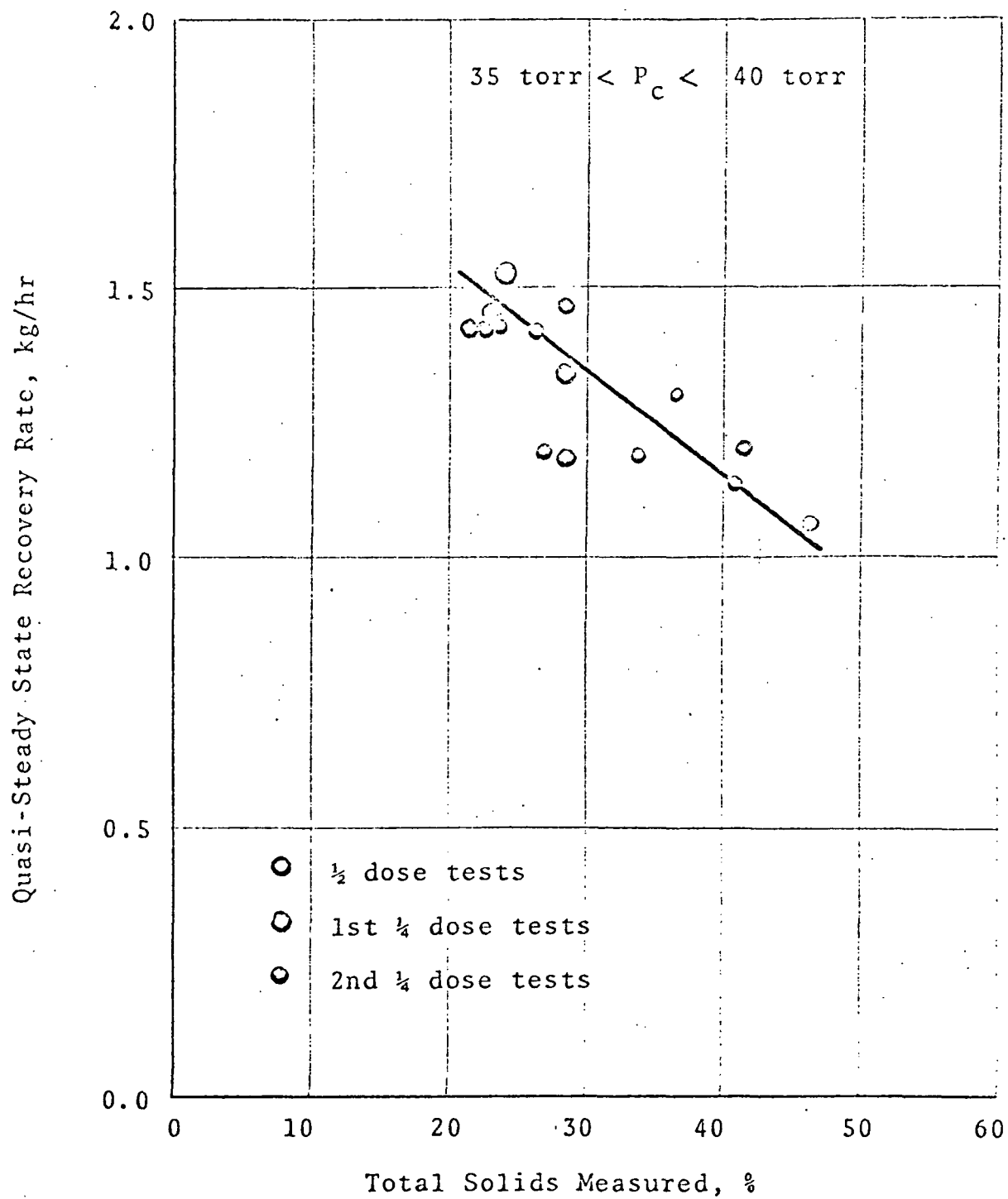
Figure 21 EFFECT OF SOLIDS CONCENTRATION ON RECOVERY RATE

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The RF was changed after each test series to avoid contamination of the recycle liquor accumulated during the next test series. Consequently, this test was unable to show that the useful life of the RF is greater than the 20 days (80 man-days) demonstrated during the first 30-day, urine-loop test.

As expected, and verified by sterility testing, the BF installed upstream of the ACF failed after 6 to 10 days of operation. It was replaced on day 15, but this time packed with a 125 gram mixture of AgCl particles and glass beads; this unit survived the remainder of the test. Thus, including AgCl particles in a BF extends its useful life from about 10 days to at least 15 days.

The BF's provided at the potable and flush water outlets were useful throughout the entire 30-day test period.

Two charcoal filters were used during the test. The first unit was on stream for 15 test days. Microbe breakthrough was detected on day 10. The unit was purposely left on stream in an attempt to saturate the bed for determination of COD loading capacity. However, saturation did not occur. The second unit was installed on day 15, along with the fresh biological filter packed with AgCl; no microbe breakthrough was detected over the remainder of the test despite the heavy microbe loads encountered on days 15 and 16 when untreated urine was distilled. The ACF's used during this test each contained 160 grams of charcoal.

The first bed was challenged with a total COD load of 11.8 grams; during the 14 days the total COD leakage (COD not removed) amounted to 3.2 grams for a 73% COD removal efficiency. The loading factor realized was 0.074 g COD/g charcoal. The bed provided an average influent-effluent COD ratio of 3.5 to 1. However, on several days (7, 9 and 14) this ratio was very nearly 1. The maximum ratio experienced was 12:1 on day 12.

The above loading number may have been adversely affected by the test sequence. The recovery rate and consequently charcoal influent flow rate declined by about 20% from day 1 to day 7 but on day 8 the flow rate returned to its initial value. The abrupt return to a flow rate 20% higher than previously may have caused some COD unloading.

Reliable loading and useful life data was not obtained for the second ACF because the quality of the raw condensate on days 15 through 19 was grossly affected by operating with untreated urine on days 15 and 16.

The AgCl column used during this test was the one used during the two previous 30-day tests. Since it performed as required this test demonstrated that it's useful life is greater than 90 days (120 + 73 + 120, or 313 man-days).

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One I-X was used for the entire test. It's performance was outstanding for the first 14 days - as shown in the next subsection. However, since it's design capacity was 200 milliequivalents of cationic and anionic species, and 760 milliequivalents of ammonia were introduced as the result of using untreated urine on day 15, the I-X was expended on day 15. Since the test time remaining was no greater than the demonstrated useful life the expended I-X was left in the system to evaluate it's silver removal capacity. As shown in the next subsection, it retained this capability at least through day 20. The explanation for this result is the displacement of ammonia from the resin by the metallic ion. Generally, the affinity of a resin type for an ionic species increases with species atomic weight. This is a favorable failure-mode since "bad actor" metals will be retained at the expense of releasing undesirable, but less harmful ionic species.

The data obtained near the end of the last test series is useful in predicting the required storage volume for recycle liquor onboard a manned spacecraft. As shown in Table 11, 2159 + 1170, or 3329 grams of recycle liquor had to be dumped over an 8-day period to maintain a fairly stable recovery rate. Since this liquor had a specific gravity near 1.17, this test indicates that the LA specific volume required for disposing of urine solids is 3329/4 x 8 x 1.17, or 89 ml/man-day (5.43 in^3/man-day).

7.3.2.3 Water Quality

Extensive chemical and biological analyses were performed on the raw condensate, ACF effluent, AgCl effluent (flush water), and I-X effluent (potable water). In addition, the recycle liquor was analyzed daily for sterility and total solids.

The quality of the raw condensate was very good - except for days 15 and 16 when no PS was used. Table 12 shows that on these days the raw condensate contained enormous quantities of ammonia. In addition, substantial biological contamination resulted from the lack of PS in the feed liquor. Thus, this test demonstrated that the PS is absolutely necessary to avoid extensive post-treatment. It also demonstrated that if the distillation unit is started and then operated for 2 days without any pretreatment, at least 2 days of operation with PS is required to avoid excessive loads on the post-treatment filters. Of course, if the recycle loop had contained concentrated, treated urine at the start of day 15, instead of deionized water, the recovery period would have been shorter.

The reason for the high COD on day 12 is unknown. It cannot be due to carryover because the ammonia concentration was low. The best explanation appears to be grease from the compressor.

C   H   E   M   T   R   I   C

Table 12   RAW CONDENSATE QUALITY DURING PS REDUCTION TEST

Test Day No.	Biopal Dose Level	Aerobic Count (#/100 ml)	Sterile	Specific Resistance (Kohm-cm)	pH	NH_3 (ppm)	COD (ppm)	Odor	Appearance
1	1/2	0	Yes	29.2	4.3	0.5	61	Slight	Clear
2	"	--	--	--	--	--	--	---	---
3	"	--	--	--	--	--	--	---	---
4	"	0	Yes	27.6	4.4	0.5	81	Slight	Clear
5	"	0	Yes	28.3	4.1	0.6	92	Slight	Clear
6	"	0	Yes	27.5	4.2	0.5	116	Slight	Clear
7	"	0	Yes	28.5	4.1	0.5	52	Slight	Clear
8	1/4	0	Yes	14.6	5.2	0.7	48	Slight	Clear
9	"	0	Yes	29.0	4.1	0.5	32	Slight	Clear
10	"	0	Yes	21.0	4.0	0.5	42	Slight	Clear
11	"	--	--	--	--	--	--	---	---
12	"	0	Yes	27.0	4.1	0.5	534	Moderate	Cloudy
13	"	0	Yes	21.0	4.0	0.5	50	Slight	Clear
14	"	0	Yes	27.0	4.1	0.7	58	Slight	Clear
15	0	0	Yes	0.6	9.4	2×10^3	190	Strong	Clear
16	"	3000	No	0.7	9.3	2×10^3	152	Strong	Cloudy
17	1/4	150	No	1.5	9.2	255	89	Strong	Cloudy
18	"	19	No	1.6	8.9	255	88	Strong	Cloudy
19	"	--	--	--	--	--	--	---	---
20	1/4	--	--	--	--	--	--	---	---
21	"	--	--	--	--	--	--	---	---
22	"	0	Yes	36.3	4.9	0.8	83	Slight	Clear
23	"	--	--	--	--	--	--	---	---
24	"	0	Yes	39.8	4.4	1.5	21	Slight	Clear
25	"	1	No	35.0	4.4	1.6	29	Slight	Clear
26	"	0	Yes	32.0	4.4	2.1	88	Slight	Clear
27	"	--	--	--	--	--	--	---	---
28	"	--	--	23.0	4.1	2.2	50	Slight	Clear
29	"	0	Yes	58.0	5.9	2.5	61	Slight	Clear
30	"	0	Yes	32.0	4.9	3.6	58	Slight	Clear

From the results of this test it is concluded that the 1/4-dose level is acceptable from the standpoint of distillation unit operation. However, the slightly higher ammonia concentrations detected on days 24 through 30 indicates that a little more sulfuric acid in the PS formulation may be beneficial.

The quality of the ACF effluent was monitored primarily to evaluate the performance of the ACF - since small changes in the PS dose level have a negligible effect on raw condensate quality. Comparison of the data presented in Tables 12 and 13 for the first fifteen days shows that the ACF increased the pH, and decreased the COD of the recovered water - even though on day 10 the ACF started to "dump" bacteria. Failure of the BF, of course, was anticipated, based on previous experience. The BF and ACF were not replaced on the 10th day so that the chemical performance of the ACF could be more accurately evaluated. Since it demonstrated a useful "chemical" life of 15 days, and 15 days remained for testing, it was replaced at the end of the 15th day along with a new BF which contained AgCl particles.

Table 13 shows that with the new BF the second ACF had a useful "biological" life of at least 15 days. However, the useful chemical life of the second ACF was dramatically shortened to one day or less by using untreated urine on the 15th and 16th days.

This test dramatically demonstrates the importance of monitoring the specific resistance or pH of the raw condensate, to avoid premature failure of the ACF. If the raw condensate on day 15 and 16 had been redistilled until it's specific resistance was above 20,000 ohm-cm, the ACF would not have been exhausted on that day. On the other hand, if COD had been monitored, raw condensate would have been unnecessarily redistilled on day 12.

Flush water (AgCl effluent) was not analyzed in detail like the raw condensate and ACF effluent - because it's composition should always be very similar to that of the ACF effluent. Flush water was monitored primarily for sterility; samples were taken aseptically on test days 11 through 18, and periodically thereafter. On each of these days the flush water was sterile. To determine the effects of the poor chemical quality of the ACF effluent on the AgCl column, flush water samples were shipped to the NASA MSC for selected analyses. The results of these analyses are presented in Table 14.

The interesting result of the flush water analyses is the effect of ammonia on the solubility of silver chloride. As shown in Table 14, the silver content was extremely high after days 15 and 16, when untreated urine was processed and the ACF was overloaded. Thus, when the AgCl influent contains a large amount of contaminant like ammonia the effluent will contain substantially more than the saturated concentration of silver and chloride ions.

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Table 13 ACF EFFLUENT QUALITY DURING PS REDUCTION TEST

Test Day No.	Biopal Dose Level	Aerobic Count (#/100 ml)	Sterile	Specific Resistance (Kohm-cm)	pH	NH_3 (ppm)	COD (ppm)	Odor	Appearance
1	1/2	0	Yes	--	--	--	--	---	---
2	"	--	--	--	--	--	--	---	---
3	"	--	--	--	--	--	--	---	---
4	"	0	Yes	28.1	5.8	0.5	16	None	Clear
5	"	0	Yes	28.6	5.6	0.3	14	None	Clear
6	"	0	Yes	25.0	5.8	0.2	40	None	Clear
7	"	0	Yes	32.0	6.8	0.4	28	None	Clear
8	1/4	0	Yes	32.5	6.2	0.9	28	None	Clear
9	"	0	Yes	28.0	6.1	1.1	36	None	Clear
10	"	30,000	No	26.0	6.0	1.1	20	None	Cloudy
11	"	--	--	--	--	--	--	---	---
12	"	2.9×10^5	No	24.5	6.8	0.4	44	None	Cloudy
13	"	1.9×10^5	No	27.0	5.6	0.2	32	None	Cloudy
14	"	1.9×10^5	No	20.0	6.7	0.3	60	None	Cloudy
15	0	0	Yes	--	--	--	--	---	---
16	"	0	Yes	0.5	9.1	1800	42	Strong	Cloudy
17	1/4	0	Yes	1.3	9.3	350	34	Strong	Cloudy
18	"	0	Yes	1.7	9.1	232	54	Strong	Cloudy
19	"	--	--	--	--	--	--	---	---
20	1/4	--	--	--	--	--	--	---	---
21	"	--	--	--	--	--	--	---	---
22	"	0	Yes	36.3	4.9	0.8	58	Slight	Clear
23	"	--	--	--	--	--	--	---	---
24	"	0	Yes	33.0	7.4	1.8	28	Slight	Clear
25	"	0	Yes	34.0	7.0	1.8	52	Slight	Clear
26	"	0	Yes	39.0	6.7	1.8	104	Slight	Clear
27	"	--	--	--	--	--	--	---	---
28	"	--	--	16.0	6.5	3.4	60	Slight	Clear
29	"	0	Yes	24.0	6.7	2.2	104	Slight	Clear
30	"	0	Yes	26.0	6.6	3.1	58	Slight	Clear

Table 14 FLUSH WATER ANALYSES DURING PS REDUCTION TEST

Test Day No.	Iron (ppm)	Nickel (ppm)	Silver (ppm)	Zinc (ppm)	Organic Carbon (ppm)
13	0.1	0.1	0.08	0.01	51
14	0.14	0.1	0.20	0.01	25
17	0.1	0.1	100.0	0.01	51
18	0.1	0.1	30.0	0.01	50
24	0.1	0.1	0.1	0.01	21
26	0.1	0.1	0.28	0.01	38

The potable water collected during this test was exceptionally pure until several days after untreated urine was processed and the ACF upstream of the I-X was expended. As shown in Table 15, the conductivity, pH, ammonia content and COD all increased dramatically after day 17. Thus, even though untreated urine was processed on days 15 and 16, and the ACF was expended on day 16 (see Table 13), the I-X was not expended until after day 17.

Even though the quality of the potable water collected after day 17 was inferior to that collected previously, it still had reasonably good resistance, pH and COD - and it was sterile. The two yeast and/or mold colonies denoted for the 24th day are attributed to sample contamination - because they were not detected until after 8 days of incubation, the I-X influent was sterile, and all subsequent samples were sterile.

7.3.2.4 Recalibration

On the day after the 30th test day the distillation unit was recalibrated once again to determine if it's performance had been degraded after 90 days of operation. For this test, tap water was fed into the unit, and the excess feed was discharged into a sewer. Figure 22 shows the integrated quantity of condensate collected. By comparing the computed average recovery rate to the pretest data presented in Figure 13 (page 4-4), it can be seen that the distillation rate, if anything, had improved. This increase in performance is attributed to the use of grease (Texaco's Unitemp 500) in reducing internal clearances in the compressor.

The range of condenser pressures measured during this test are presented in Figure 22 to illustrate how this parameter varies during a typical test. In the morning, after being in a stand-by mode overnight, condenser pressure is near the saturation pressure of water at room temperature. After start-up, it asymptotically increases to near 40 torr - unless the room temperature decreases as it did during this recalibration test.

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Table 15 POTABLE WATER QUALITY DURING PS REDUCTION TEST

Test Day No.	Biopal Dose Level	Biological Count (no./100 ml)				Specific Resistance (Kohm-cm)	pH	NH_3 (ppm)	COD (ppm)
		Aerobic	Anerobic	Coliform	Yst & Mld				
1	1/2	0	0	0	0	180	5.4	0.2	20
2	"	-	-	-	-	-	-	-	-
3	"	-	-	-	-	-	-	-	-
4	"	0	0	0	0	210	5.9	0.2	14
5	"	0	0	0	0	210	6.5	0.2	15
6	"	0	0	0	0	130	6.5	0.1	16
7	"	0	0	0	0	110	6.9	0.2	36
8	1/4	0	0	0	0	125	6.9	0.2	22
9	"	0	0	0	0	190	6.3	0.2	26
10	"	0	0	0	0	190	6.3	0.2	30
11	"	-	-	-	-	250	6.4	0.2	19
12	"	0	0	0	0	250	5.6	0.2	38
13	"	0	0	0	0	240	5.7	0.2	96
14	"	0	0	0	0	310	6.1	0.2	80
15	0	0	0	0	0	260	5.9	0.3	69
16	"	0	0	0	0	250	5.9	0.2	50
17	1/4	0	0	0	0	250	5.6	0.6	44
18	"	0	0	0	0	-	-	-	-
19	"	-	-	-	-	13	6.9	10.6	100
20	1/4	-	-	-	-	14	6.0	9.8	80
21	"	-	-	-	-	19	5.1	7.5	60
22	"	0	0	0	0	18	6.0	7.5	58
23	"	-	-	-	-	24	7.1	6.2	60
24	"	0	0	0	2	25	7.0	5.1	30
25	"	0	0	0	0	22	6.1	3.2	49
26	"	0	0	0	0	33	6.9	4.0	88
27	"	0	0	0	0	21	7.0	3.0	90
28	"	-	-	-	-	24	7.1	2.0	80
29	"	0	0	0	0	22	6.9	2.0	61
30	"	0	0	0	0	20	7.0	2.0	78
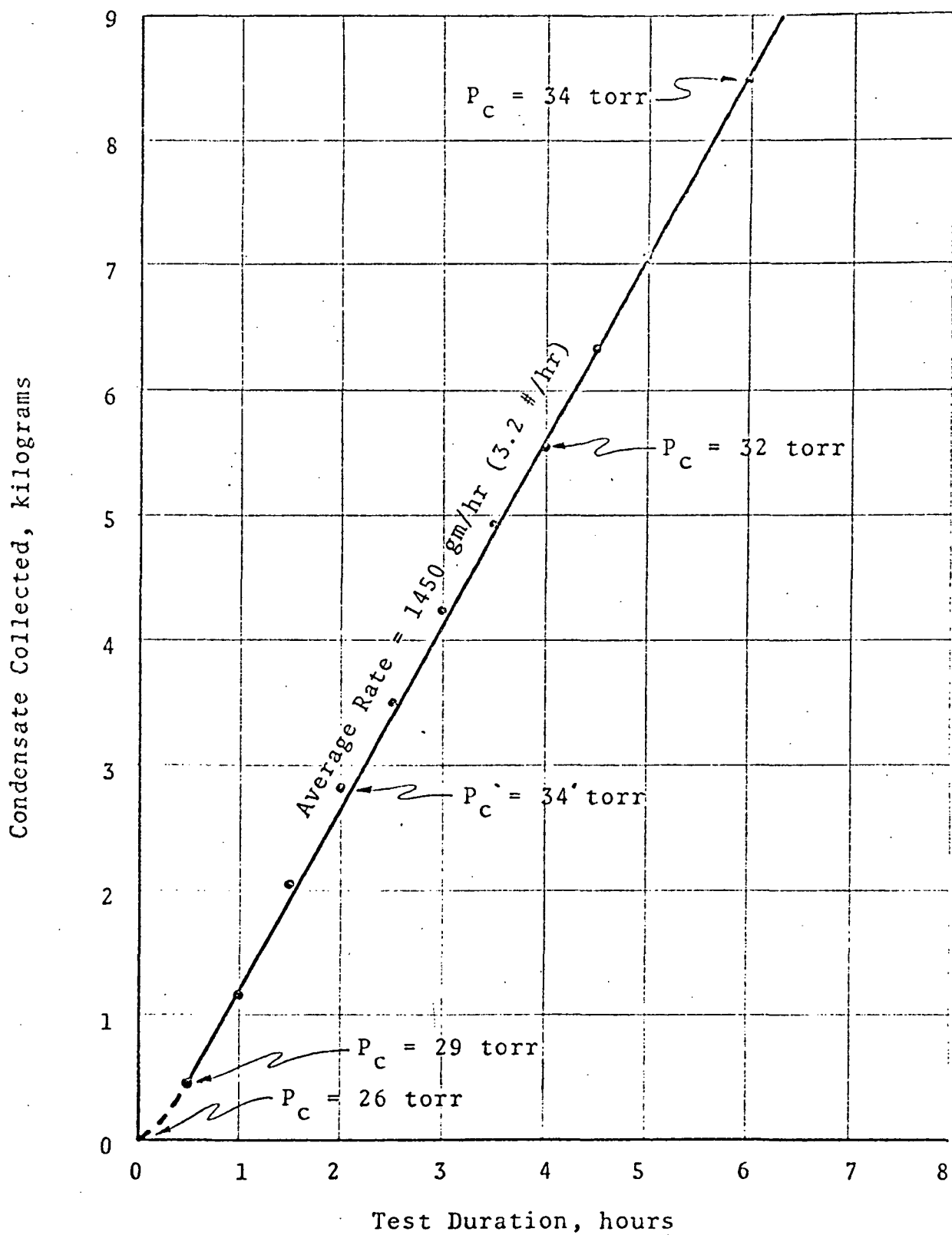
Figure 22 DISTILLATION RATE AT END OF PS REDUCTION TEST

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7.3.3 Conclusions & Recommendations

The PS reduction tests achieved their primary objective - namely, "to determine the minimum quantity of PS required" when processing urine and urinal flush water in a compression distillation unit. The secondary objective was also achieved - namely, "to extend the useful life of the activated carbon filter used to post-treat distillate". In addition, this test demonstrated that the potable water produced will be sterile if the deionizer is prepasteurized.

The static tests demonstrated that under normal conditions, the quantity of Biopal used in the PS formulation can be reduced to 1/4th the amount used in the first 30-day, urine-loop tests. However, this quantity is marginal if the quantity of urine collected during each micturation is twice the normal quantity. Both 1/2 and 1/4 Biopal dose levels were evaluated during the urine-loop test; the results indicate that the thermodynamic performance of the distillation unit, and the quality of the raw condensate are essentially the same at both dose levels. Therefore, it is concluded that the 1/4 dose level should be used if a safety factor of about 2 is considered adequate; if a larger margin is desired, a correspondingly higher dose level should be used.

This 30-day, urine-loop test demonstrated that the useful biological life of a BF and ACF subassembly can be extended to at least 15 days by placing AgCl particles and glass beads in the BF. This test also demonstrated that the useful biological life of a deionizer is at least 17 days - if it is prepasteurized and the influent water is saturated with silver chloride ions. Both of these improvements should be employed on all future tests with water recovery systems.

These tests also yielded a substantial amount of new design and operating data; namely:

1. The evaporator surface in the distillation unit is easily fouled with precipitated solids if the feed liquor does not contain the developed PS.

2. The coupling in the distillation unit can be assembled so that it experiences excessive loads; it should be redesigned to avoid this problem.

3. When untreated urine is processed the condenser purge rate must be increased to dispose of the non-condensable gases produced by putrification and/or fermentation.

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4. The specific loading attainable with the ACF is 0.074 g COD/g charcoal, and the dynamic removal efficiency is 73%.

5. The AgCl has a useful life greater than 313 man-days.

6. The LA volume required is 89 ml/man-day.

7. The PS formulated may be improved slightly by containing more sulfuric acid.

8. The ACF has a useful chemical life greater than 60 man-days.

9. Overloading the post-treatment filters can easily be avoided by monitoring the specific resistance and/or pH of the raw condensate, and recycling the raw condensate whenever these parameters are out-of-spec.

10. The flush water will be dosed with very large quantities of silver ions if the influent to the AgCl column contains large amounts of ammonia.

HIGH-SOLIDS TEST

During the 30-day urine-loop and wash-loop tests, and the pretreatment reduction tests, the concentration of solids in the recycle liquor was limited to approximately 30% by periodically bleeding-off (dumping) part of the recycle liquor. This solids level was selected because previous experience had shown that compression distillation units can reliably process this type of liquor, and still produce high quality condensate with a very low specific energy demand. Higher water yields could have been attained by allowing the solids concentration to exceed 30% - but this was not done because the primary objective of the first loop tests was to demonstrate reliable operation.

Since the distillation unit had performed remarkably well during the first 30-day, urine-loop test - it was operated for several more days after that test without dumping recycle liquor, to determine how high the solids concentration could be before operational problems were encountered (see Section 5.2.4). These cursory tests indicated that the upper limit is near 64% total solids. As a result of these tests, and the continuing interest in reducing the quantity of make-up water required on a space station, contract NAS9-9191 was modified to include another 30-day, urine-loop test; the specific objective of this test was to determine (1) the effect of solids concentration on water quality, and (2) the maximum concentration which permits stable operation. The following subsections indicate that these objectives were achieved.

8.1 Test Set-Up and Procedure

This test was performed using essentially the same equipment and arrangement used for the pretreatment reduction tests (see Section 7.1). However, as shown in Figure 23, a bladderless tank was installed in the recycle loop. The purpose of this tank was to increase the volume of the recycle loop so that (1) daily variations in solids concentration would be minimized and (2) the average concentration of solids would increase at a relatively slow rate over a 30-day period.

The required size of the recycle tank was computed as follows.

A. Desired maximum increase in solids concentration during each test day was 3%.

B. Maximum concentration of dissolved solids anticipated in treated urine and urine flush water was 3%.

C. Volume of treated urine and urinal flush water anticipated each day was (250 + 150) x 6 x 4 = 9600 ml.

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Figure 23   FLOW SCHEMATIC FOR HIGH SOLIDS TEST

D. Therefore, the required volume of the recycle loop is 9600 x 3/3, or 9600 ml.

E. Since the approximate volume of all other components in the recycle loop is 1700 ml, the required minimum volume of the recycle tank is 9600 - 1700, or 7900 ml.

Since the two double-bladder tanks furnished with the hardware each have a capacity of 8.4 liters, and only one was being used for accummulating waste water and flush water, the other tank was installed as the recycle tank (without the bladders).

The 1/4-dose PS formulation established for the previous tests was used in this test (see page 7-9). Previous urine collection and PS dosing practices were duplicated. Large (20 day) batches of the PS formulation were prepared so that PS stability could be verified. The total weight and average specific gravity of each day's input was measured.

Since the primary objective of this test was to determine the maximum acceptable concentration of solids in the recycle loop, liquor was not "dumped" from the recycle loop until the later half of the test - and then, only for the purpose of obtaining a sample for total solids determinations. During the first half of the test, solids concentration was estimated by (1) measuring the specific gravity of a sample which was returned to the recycle loop, and (2) using a previously developed correlation between the specific gravity of urine recycle liquor and total solids. The applicability of this correlation was reverified during the later half of the test when both specific gravity and total solids data was obtained. The mass and specific gravity of the daily input was also used to estimate the concentration of solids in the recycle loop.

As in previous tests, water quality was monitored. Reduced emphasis was placed on biological analyses because of the previously demonstrated performance of the silver column and the effective decontamination procedures applied to the deionizer. However, sufficient monitoring was performed to adequately assess the biological purity of the product water at each processing stage. Chemical analyses were emphasized, and in particular, analyses directed to the assessment of the concentration of organic contamination. In addition to shipping samples of water to the NASA MSC for analysis, samples were also shipped to the Nalco Chemical Company for TOC analysis.

Extensive chemical analyses were undertaken to identify the impact of high-solids operation on expendable components in the post-treatment loop. The post-treatment components in the loop at the end of the previous tests were used unaltered in this test during the initial test period. Later in the test a fresh charcoal column and deionizer were installed.

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8.2 Results & Discussion

The urine loop operated for 48 consecutive days, plus one day for recalibration of the distillation unit. More than 30 days of operation was required to determine the maximum acceptable solids concentration because (1) the solids content of the treated urine and urinal flush water was less than 3%, (2) some of the solids precipitated in the recycle loop, and (3) some of the solids were removed from the recycle loop as the result of a filter change and taking samples for total solids determinations. The test was terminated at the end of the 48th day because the total solids concentration was above 50%, and the recycle liquor appeared to be rapidly becoming too viscous to withdraw from the evaporator.

During this test the distillation unit did not experience any mechanical failures; however, two maintenance operations and a procedural change were necessary.

On the 27th, 31st and 32nd days the recycle flow became erratic and finally stopped. The problem was diagnosed as excessive liquor viscosity resulting from over concentration of the evaporator's contents during the end of the previous day's test. Recycle flow was restarted by back flushing the internal recycle passageways with tap water, to dilute the liquor in the evaporator sump. To avoid a reoccurrence of this problem, the daily shut-down procedure was changed on the 33rd day so that (1) the evaporator is flushed with raw condensate after the feed tank is emptied, and (2) the drive motors are not stopped until after the delta P increases to about 15 torr, which indicates that essentially all of the liquor has been removed from the evaporator.

On the 39th day at approximately the mid-point in the run, the power began to increase and in a short time had increased by 30 watts. The power increase was attributed to either the beginning of motor failure or the need for timing gear lubrication. At the end of the run, the motor winding resistances were measured and found to be normal. The timing gears were then relubricated without removing the motor. Power consumption returned to normal the next day and remained normal during the remaining test days. The timing gears had been lubricated in preparation for running the Pretreatment Reduction Test, and had not been serviced since that time. The resultant lubrication interval amounts to 70 days (68 days with waste and 2 days of calibration with tap water).

Unscheduled, but anticipated, maintenance was limited to a one-time replacement of the following items.

A. Recycle Filter
B. Activated Carbon Filter

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C. Deionizer
D. BF at potable outlet
E. BF at flush outlet

All tubing in the peristaltic pump was replaced at the start of the test, and on the 18th and 40th days; the recycle pump tubing was also replaced on the 22nd and 44th days.

On the 43rd day a small section of 1/4" O.D. tubing in the recycle loop (downstream of the recycle tank) was plugged with precipitated solids. After flushing with tap water it remained open for the remainder of the test.

8.2.1 Thermodynamic Performance

The daily inputs and outputs, and the average steady-state recovery rates and specific energy demands for the 48-day, high-solids test are summarized in Table 16. The results obtained, and the anomalies and trends in the data are described as follows.

1. The daily input ranged from 7,701 to 11,285 grams, depending upon the quantity of urine collected. Since 152.25 x 24 x 48, or 175,392 grams of the total input was treated flush water, and 1200 + 500 + 2000 + 200, or 3,900 grams was tap water, the quantity of urine processed was 427,392 - or 248,223 grams. Thus, the average mass of urine collected was 248,223/24 x 48, or 215 grams/micturation. This is 277 - 215, or 62 grams/micturation less than the average mass of urine collected during the 30-day, urine-loop test. The explanation for this is the effect of relative humidity on the quantity of body water excreted as urine; the first 30-day, urine-loop test was performed during the months of August and September when the relative humidity was in the range of 50-60% - while the high-solids test was performed in February and March when the RH ranged from 30 to 40%.

2. The median concentration of solids in the waste feed was 1.9%. Thus, the median quantity of urine solids collected was (152.25 + 215) 0.019 - 1.12*, or 5.77 grams/micturation - or 5.77 x 6, or 34.62 grams/man-day. Since the amount of urine solids excreted each day is usually in the range of 50 to 60 grams/man-day, it is concluded that the urine collected each day did not include a sufficient number of "early morning" micturations, and/or many of the donors drank excessive amounts of coffee.

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*Solids in PS (see page 7-9)

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Table 16 - PERFORMANCE SUMMARY FOR HIGH SOLIDS TEST

Test Day No.	Waste Input Total Grams	Waste Input % Solids	Water Output (grams)	Purge Loss (grams)	Liquor Dumped (grams) Water	Liquor Dumped (grams) Solids	Liquor Comp. (% solids) Calc.	Liquor Comp. (% solids) Meas.	Recovery Rate (kg/hr)	Specific Energy (w-h/kg)
1	8373	2.1	7978	61	0	0	1.7	-	a	a
2	8269	2.1	8245	60	0	0	3.5	-	a	a
3	9618	2.2	9227	96	0	0	5.5	-	1.55	55
4	8741	1.8	8345	63	0	0	6.9	-	1.54	55
5	8719	1.9	8293	66	0	0	8.5	-	1.53	55
6	8849	2.3	8395	63	0	0	10.3	-	1.48	55
7	9022	1.8	9131	69	0	0	11.7	-	1.49	57
8	9864	2.0	9488	112	0	0	13.5	-	b	b
9	10001	2.0	10030	74	0	0	15.2	-	b	b
10	8513	2.0	8285	95	0	0	16.2	-	1.37	62
11	8903	2.0	8760	100	0	0	18.1	-	1.37	63
12	9524	1.9	9357	75	0	0	19.6	-	1.41	63
13	8393	1.9	8198	68	0	0	21.0	-	1.36	65
14	9386	1.9	9351	73	0	0	22.3	-	1.35	66
15	8782	1.9	8567	111	0	0	23.7	-	1.32	68
16	8446	1.9	8370	90	0	0	25.0	-	1.29	65
17	8741	1.9	8282	107	0	0	26.2	-	1.26	71
18	7765	2.0	7285	100	0	0	27.4	-	1.28	75
19	9288	1.7	9083	96	0	0	28.7	-	1.33	70
20	8115	2.0	8070	90	0	0	30.0	-	1.53	71
21	8273	1.9	7995	105	0	0	31.2	-	1.32	71
22	8880	2.0	8561	115	0	0	32.6	-	1.35	74
23	8866	2.1	8635	94	0	0	34.2	-	1.27	73
24	8429	2.0	8112	106	0	0	35.4	-	1.27	74
25	9196	1.7	8954	111	138	62	36.6	30.8	1.26	75
26	8277	1.8	7929	103	0	0	37.2	-	1.24	77
27	9498c	1.5	9015	120	345	201	38.2	36.8	1.21	81

(continued on next page)

(a) Run too short to achieve Pc $\geq$ 35 torr during test.
(b) Room temperature too low to achieve Pc $\geq$ 35 torr during test.
(c) Includes 1200 grams of tap water.

(continuation of Table 16)

Test Day No.	Waste Input Total Grams	% Solids	Water Output (grams)	Purge Loss (grams)	Liquor Dumped (grams) Water	Solids	Liquor Comp. (% solids) Calc.	Meas.	Recovery Rate (kg/hr)	Specific Energy (w-h/kg)
28	8071	1.8	7750	115	0	0	37.8	-	1.23	77
29	7701	1.4	7505	130	0	0	38.4	-	1.20	80
30	9711	1.7	9224	136	0	0	39.7	-	1.18	80
31	9417d	1.6	9302	157	0	0	40.7	-	1.11	86
32	11285e	1.6	11840	275	422	164g	42.0	-	1.07	90
33	8982	1.9	8640	160	0	0	42.0	-	0.97h	96h
34	8658	1.8	7688	150	0	0	43.5	-	1.11	87
35	8936	1.9	9508	204	152	88	44.3	36.9	0.92	102
36	9162	1.9	8775	195	0	0	44.7	-	0.92	107
37	10359	1.9	10150	200	0	0	46.4	-	0.90	102
38	8707	1.8	7830	160	0	0	47.5	-	0.96	104
39	8700	1.9	8585	190	0	0	48.9	-	0.92	109
40	9198	2.1	8775	·162	0	0	50.1	-	0.96	101
41	9137	1.8	8937	160	0	0	50.8	-	0.86	122
42	8704	2.2	8330	172	0	0	52.0	-	i	i
43	9387f	1.5	8285	·145	0	0	54.2	-	i	i
44	7791	1.7	7530	150	343	270	55.0	44.1	j	j
45	9031	2.1	9417	200	0	0	52.0	-	0.90	110
46	8712	2.0	8245	80	0	0	53.4	-	0.88	112
47	8856	2.2	8130	165	0	0	55.0	-	0.83	119
48	8279	1.9	8145	166	0	0	56.1	46.7	0.82	119
Σ	427515		414532	5896	1400	785				

(d) Includes 500 grams of tap water
(e) Includes 2000 grams of tap water
(f) Includes 200 grams of tap water
(g) Sample taken from changed-out recycle filter
(h) Evaporator flushed with raw condensate at end of run
(i) Feed line plugged - recovery rate erratic
(j) Recycle tubing split - lost ~ 450 gms of liquor

3. Purge losses increased during the test from less than 1% to near 2%. This trend is partially explained by the need for increased running time and higher average condenser pressure as solids were accumulated in the recycle loop. Since the purge losses on the 46th day were also less than 1% it is concluded that the test operators tended to overpurge the distillation unit as delta P increased due to the accumulation of solids in the recycle loop.

4. The measured concentrations of total solids in samples of the recycle liquor were always less than the calculated concentrations. This is attributed to precipitation and the accumulation of solids within the recyle loop - as evidenced by precipitated solids found in the recycle tank after the test was completed.

8.2.1.1 Recovery Rate

Table 16 shows that the recovery rate (average) decreased from about 1.6 kg/hour to 0.82 kg/hour over the test period. The downward trend was fairly smooth - except for several days on which the recovery rate increased as much as 14%. These anomalies are illustrated in Figure 24, and explained as follows.

The distillation unit is provided with a recycle loop to (1) aid in the disposal of solids, (2) prevent the accumulation of solids on the evaporation surface, and (3) maximize the effectiveness of the evaporator by preventing gross variations in the solids concentration over the evaporator surface. This test dramatically demonstrated the significance of the last two reasons.

Initially, the feed valve was adjusted to obtain a rotameter reading of 100, which corresponds to a water flow rate of about 6.0 kg/hour. However, as solids were accumulated in the recycle loop the feed liquor became more viscous, causing the rotameter to read higher - unless the feed valve setting was reduced, as was done by the test crew to maintain a reading of 100. Thus, the feed rate was inadvertently reduced as the test progressed. Finally, on the 18th day when the recycle flow appeared to be "sluggish", the feed valve opening was increased to obtain a rotameter reading of 125. As shown in Figure 24, this action caused the average recovery rate to increase from about 1.26 kg/hour to 1.33 kg/hour. The feed valve opening was increased again on days 23 and 40, when the recycle flow again appeared to be sluggish. From the results obtained it is obvious that if the feed-to-recovery rate ratio is too low the average recovery rate is unnecessarily depressed as the result of excessive solids concentrations in the evaporator.

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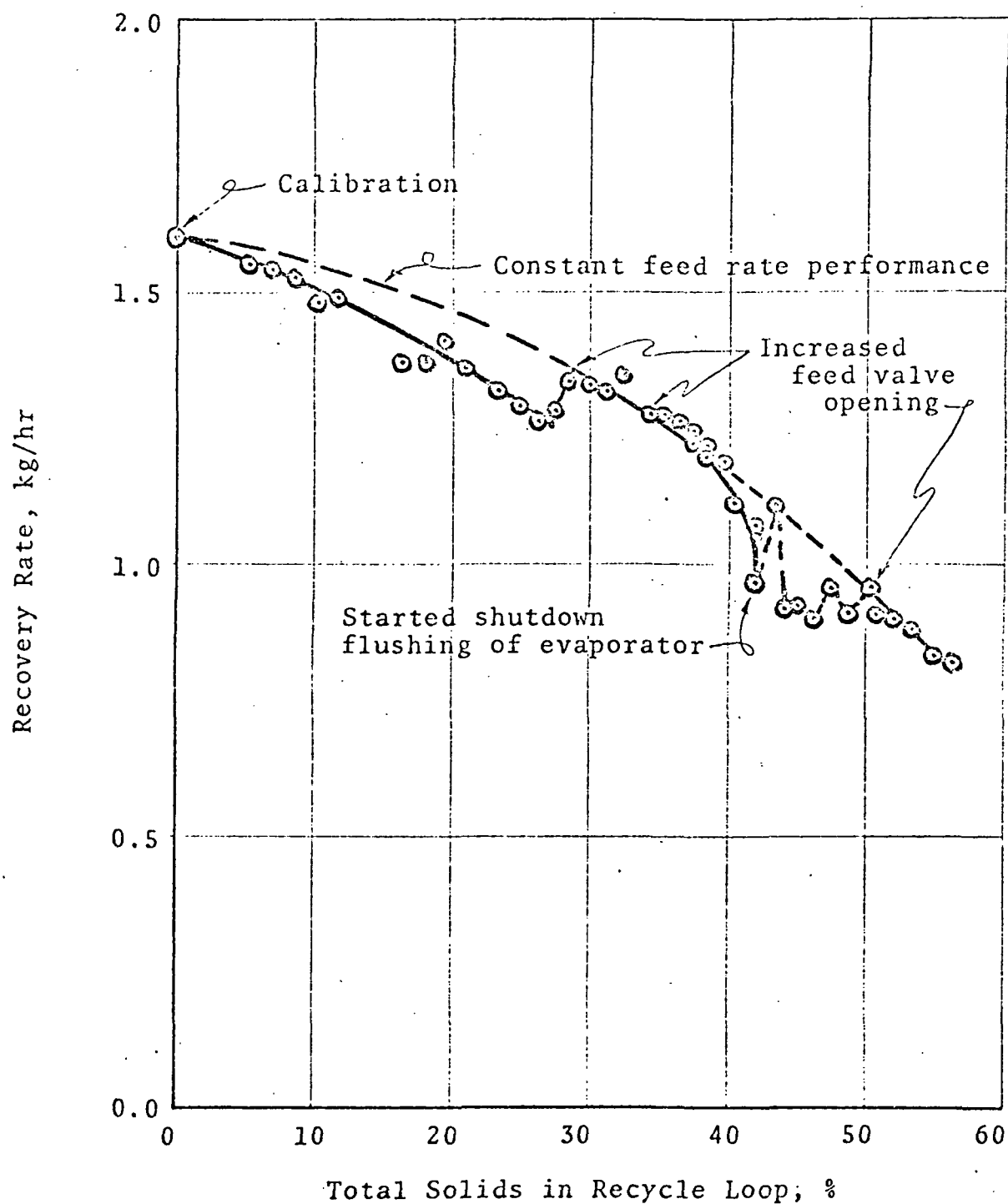
Figure 24   EFFECT OF RECYCLE SOLIDS ON RECOVERY RATE

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The effect [illegible] the evaporator with raw condensate prior to shut-down [illegible] illustrated in Figure 24. If this procedure had not [illegible] pted on the 33rd day, the test would have been terminated - and the conclusion made that the maximum allowable concentration of solids in the recycle loop is only 40%. However, by flushing the evaporator during shut-down, excessive concentrations are avoided, and the maximum allowable concentration is extended to at least 56%.

The test was intentionally terminated at the end of the 48th day because the solids concentration was above 50%, and the recycle liquor appeared to be rapidly becoming too viscous to withdraw from the evaporator. The measured concentration of solids in the sample of the recycle liquor withdrawn at the end of the 48th day was only 46.7%, even though the solids balance indicated a 56.1% concentration. The difference was due to precipitation, as evidenced by the large quantity of solids found in the recycle tank when it was finally opened for inspection. From this result it is concluded that (1) the recycle filter should be located within the recycle tank, and (2) the recycle filter/tank should be replaced before the total solids concentration reaches 56% (a safe upper limit appears to be 50%). If the penalty for electric power is relatively high, the optimum solids concentration may be less than 50%.

8.2.1.2 Water Yield

This test was performed to accurately demonstrate the water yield obtainable with a compression distillation unit which disposes of urine and PS solids by accumulating them in a recycle loop which includes a replaceable tank. The starting and ending contents of the recycle loop were accurately measured, in addition to the specific gravity and mass of the treated urine and flush water processed.

The overall mass balance was found to be as follows.

Input:	Treated Urine & Flush Water	427,515 gms
	Starting Inventory (Recycle Loop)	10,100
	Measured Total Input	437,615 gms
Output:	Condensate (Flush + Potable)	414,532 gms
	Purge Losses	5,896
	Liquor Samples, Water	1,400
	Liquor Samples, Solids	785
	Liquor Lost (Spillage)	∼450
	Ending Inventory (Recycle Loop)	14,771
	Measured Total Output	∼437,834 gms
Unaccounted Mass (Input-Output)..........		∼ -219 gms

Thus, the inputs and outputs essentially balance, even though condensate was measured in a graduated, one-liter cylinder.

The specific gravity and mass measurements made on the treated urine and flush water indicated that the input contained 8,228 grams of solids. Thus, the water available to produce condensate was 427,515 - 8,228 + 10,100* - 1,700**, or 427,687 grams - and the demonstrated water yield is 414,532 x 100/427,687, or 96.9%. Since 5,896 x 100/427,687, or 1.4% of the available water was lost in purging the condenser - 100.0 - 96.9 - 1.4, or 1.7% of the available water was used to dispose of solids - which is less than the concentration of solids in the treated urine and flush water (8,228 x 100/427,515, or 1.9%). If the test had been terminated when the solids in the recycle loop reached the 50% level, which appears to be a safe upper limit for a flight system, the available water lost would have equaled the mass of solids fed into the recycle loop. Thus, this test demonstrated that the water yield obtainable with a compression distillation unit processing treated urine is described by the following equation.

Yield $= 100 (1-C-P).$ %.

Where C $=$ concentration of solids in waste water.
P $=$ fraction lost by purging condenser.

If purge vapor is recovered (e.g., by using a purge pump and condenser) the water yield then equals $100 (1-C)$%.

8.2.1.3 Heat Transfer

The high solids test provided an excellent opportunity to not only determine the effect of solids concentration on recovery rate, but also to determine the effect of solids on the overall heat transfer coefficient (U). As described in Section 2.2.4, previously developed compression distillation units are unable to achieve U's above 75 Btu/hr-ft^2-°F (36.6 cal/hr-cm^2-°C) when processing treated or untreated urine - even though classical data for boiling water indicates that values above 150 Bty/hr-ft^2-°F (73.2 cal/hr-cm^2-°C) should be attainable. Figure 25, on the next page, indicates that the distillation unit developed under this program has a U of 120 cal/hr-cm^2-°C when processing water. However, as solids are accumulated in the recycle loop, as done during the high solids test, the U eventually decreases to around 30 cal/hr-cm^2-°C. This high performance is attributed to (1) the use of an annular dam to assure wetting

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* Starting Inventory

**Water lost in liquor samples and spillage.

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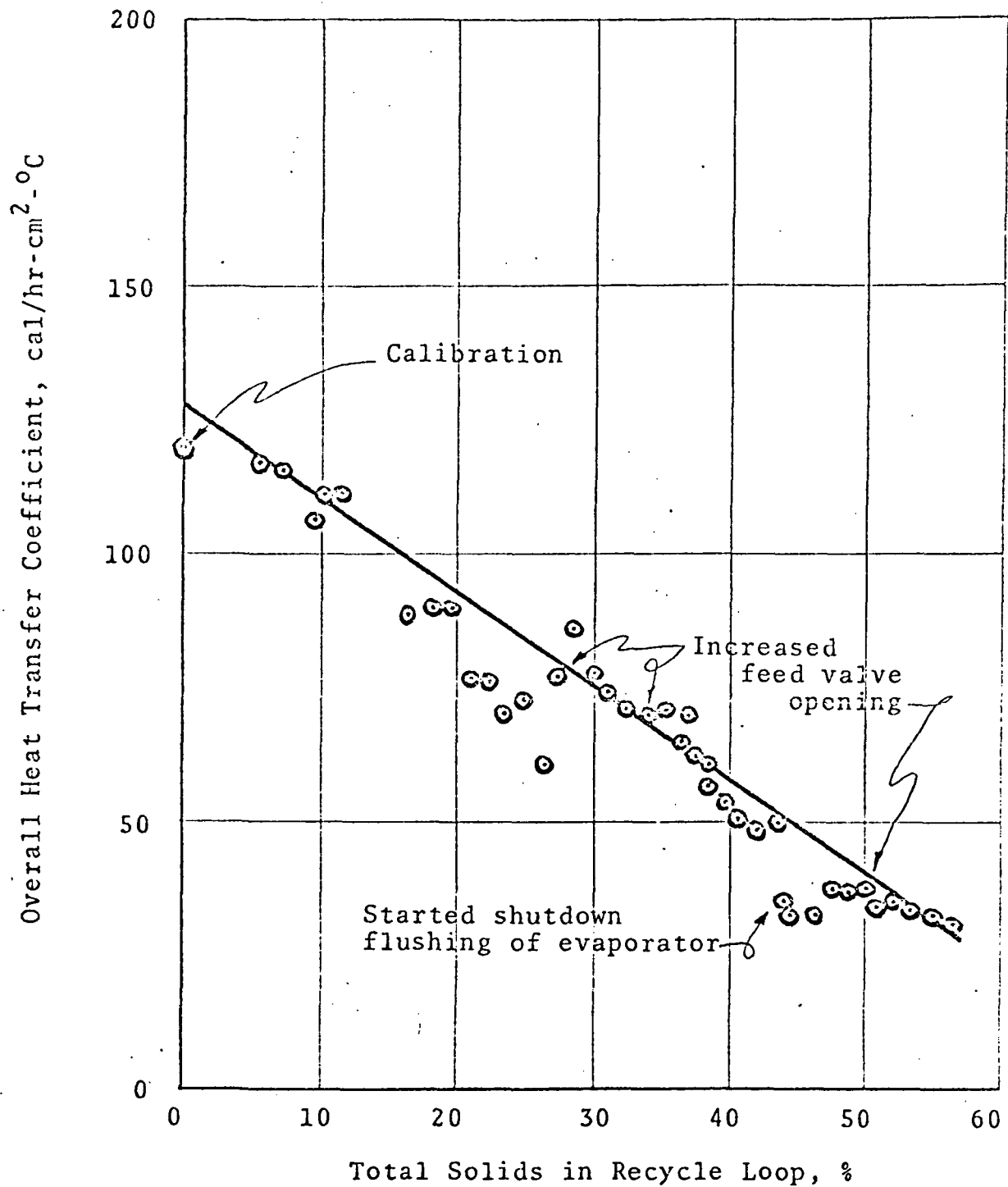
Figure 25   EFFECT OF RECYCLE SOLIDS ON HEAT TRANSFER

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of the entire evaporator surface, and (2) the high centrifugal speed which increases the film coefficient.

The overall heat transfer coefficient data plotted in Figure 25 were computed using (1) experimentally determined recovery rates, (2) the average heat of vaporization for water in the range of 30 to 35 torr, (3) the known condensation/evaporation area, and (4) the assumption that the condensing and evaporating vapors are saturated at the conditions indicated by the observed condenser and evaporator pressures. Thus, this data should not be interpreted as being film coefficients - which are substantially higher values which do not account for the effect of dissolved solids on boiling point elevation. However, this data is useful for future designs - and in gaining a better understanding of the compression distillation process.

The surprising result indicated by Figure 25 is the rather linear effect of total solids on U. This cannot be explained by boiling point elevation alone, because dissolved solids should decrease the computed U at an increasing rate. Two other factors obviously assist in producing this result - namely, precipitation and viscosity.

The abscissa in Figure 25 is "total" solids contained in the recycle loop. If dissolved solids had been used to prepare this plot, U would have decreased at an increasing rate, at least in the range of 30 to 50% solids, where most of the precipitation occurs. Thus, the linearity achieved in the 30 to 50% range is, at least, partially understandable.

The slope of the correlating line is substantially larger than anticipated. From the observed viscosity of concentrated urine, and the known effect of viscosity on film coefficients, it is concluded that the strong effect of solids on U, especially in the range of 0 to 30% solids, is due to viscosity.

8.2.1.4 Vapor Compressor

The calibrated performance of the distillation unit, as described in Section 4, is essentially as predicted by the design calculations presented in Section 2. Since the overall heat transfer coefficient is higher than anticipated, the performance of the compressor must then be lower than predicted.

The key design parameter in sizing the compressor is the slip constant (K), which relates slip rpm to pressure differential across the compressor and the specific volume of the inlet vapor. Using the correlation developed by Charanian et.al for commercially available compressors (see page 2-12), it was predicted that the new compressor would have a slip constant of 240. Table 17 on the next page shows that the value computed

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Table 17 - COMPRESSOR PERFORMANCE DATA

Test Day No.	Recovery Rate (kg/hr)	Avg. Pc (torr)	Avg. ΔP (torr)	Avg. Pc (torr)	Avg. Ve (m³/kg)	Slip Speed (rpm)	Slip Constant K
3	1.55	36	5½	30½	34.2	970	420
4	1.54	36	5	31	33.7	1020	470
5	1.53	37	6½	30½	34.2	1000	400
6	1.48	37	6	31	33.7	1110	470
7	1.49	37	6	31	33.7	1090	470
10	1.37	36	7	29	35.9	1140	430
11	1.37	37	7½	29½	35.4	1160	430
12	1.41	38	7½	30½	34.2	1170	440
13	1.36	38½	8	30½	34.2	1250	450
14	1.35	38½	8	30½	34.2	1260	460
15	1.32	38	8½	29½	35.4	1240	430
16	1.29	36	8	28	37.2	1190	410
17	1.26	38½	9	29½	35.4	1330	450
18	1.28	38	7½	30½	34.2	1360	510
19	1.33	38½	7	30½	34.2	1290	500
20	1.33	38½	7½	31	33.7	1320	500
21	1.32	38½	8	30½	34.2	1310	470
22	1.35	38	8½	29½	35.4	1200	420
23	1.27	38	8½	29½	35.4	1310	450
24	1.27	37½	8	29½	35.4	1310	470
25	1.26	38	8½	29½	35.4	1330	460
26	1.24	37	8	29	35.9	1330	470
27	1.21	38	9	29	35.9	1380	460
28	1.23	38	9	29	35.9	1350	450
29	1.20	38	9	29	35.9	1390	460
30	1.18	39	10	29	35.9	1420	450
31	1.11	38½	10	28½	36.5	1500	470
32	1.07	39	11	28	37.2	1560	460
33	0.97	39	12	27	38.5	1570	440
34	1.11	39	10	29	35.9	1670	530
35	0.92	39	12	27	38.5	1710	480
36	0.92	39½	13	26½	39.2	1690	450
37	0.90	39½	12½	27	38.5	1750	480
38	0.96	39½	11½	28½	36.5	1730	510
39	0.92	39½	11½	28½	36.5	1790	520
40	0.96	39	12	27	38.5	1650	460
41	0.86	38	11½	26½	39.2	1770	500
45	0.90	37½	12	25½	40.7	1660	450
46	0.88	39	12	27	38.5	1780	500
47	0.83	39	12	27	38.5	1860	520
48	0.82	38	12	26	39.8	1830	500

Avg. K = 465

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from the recovery rate and pressure data is 465 ± 65. Thus, at a given vapor flow rate the compressor cannot develop as high a pressure differential as anticipated. However, the design recovery rate is achieved because with the overall heat transfer coefficient attained the required pressure differentials are less than anticipated.

The discrepancy between predicted and actual slip constants is believed to be primarily due to pressure losses across the demister and the inlet and outlet ports - because the compressor lobes were coated with a lubricant to reduce internal clearances below those used in commercial practice. Thus, even higher recovery rates and lower specific energy demands should be attainable if these "orifices" are enlarged to improve compressor efficiency.

8.2.2   Expendables Required

The expendable items used in the 48-day, high-solids test were:

A.   Pretreatment Solution (PS)
B.   Recycle Filter (RF)
C.   Biological Filters (BF)
D.   Activated Carbon Filter (ACF)
E.   Silver Chloride Column (AgCl)
F.   Deionizer Column (I-X)
G.   Recycle Tank (RT)

The quantity of PS used over the 48-day period was maintained at 2.25 grams per micturation. Therefore, the total quantity used was 2.25 x 6 x 4 x 48, or 2592 grams.

The RF cartridge installed at the beginning of this test was replaced on the 32nd day because of excessive pressure drop - as evidenced by a pressure gage between the RF and the feed pump. The second cartridge remained on stream to the end of the test without any indication of plugging. Thus, this test extended the demonstrated useful life of the RF cartridge from 80 man-days (30-day, urine-loop test) to at least 30 x 4 or 120 man-days. Upon analysis, the cartridge was found to contain 60 grams of solids - which represents a very low "dirt" capacity for this type of cartridge. Because of the viscous nature of the recycle liquor, it is concluded that failure of the RF cartridge was partially due to plugging, and partially due to viscous pressure losses.

Three of the four BF's installed in the system are used as "barriers" against the migration of microbes into the condensate - namely, (1) ambient to potable, (2) ambient to flush, and (3) feed to raw condensate. These BF's were all replaced on the

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18th day, when both the flush and potable water draw-off rates were less than normal. Since these BF's were installed at the beginning of the 30-day Pretreatment Reduction Test it is concluded that their demonstrated useful life is 47 days. The BF upstream of the ACF was not changed during this test; since it was installed on the 15th day of the previous test, it's demonstrated useful life is 63 days. From this result it is concluded that the useful life of the feed-to-condensate BF is also at least 63 days - especially if it is packed with AgCl particles.

Fresh ACF and I-X columns were not installed for the start of this test - because (1) the primary biochemical objective was to determine the effect of high solids operation on the quality of the raw condensate, and (2) the ACF and I-X used to end the previous 30-day test had not exceeded their useful "biological" life. However, they were both replaced at the end of the 17th day - so that useful chemical and biological life of 31 days could be demonstrated before this test was completed. As indicated in the next subsection, this was accomplished.

The second ACF was challenged with a total of 0.057 # COD - of which 0.026 # COD were absorbed. The resultant loading factor was 0.074 #COD/# charcoal - which is lower than the value anticipated (i.e., 0.1 #COD/# charcoal). The low absorption efficiency and loading factor attained are undoubtedly due to the influent COD variations experienced - as evidenced in the next section by COD data which shows that on several days the effluent COD was higher than the influent. However, in spite of this lower than anticipated performance, the effluent COD was less than 100 ppm on all 31 days tested, except for the 46th day when the ACF effluent COD was 110 ppm.

The I-X was designed for 200 milliequivalent static capacity - i.e., 720 grains of NaCl under equilibrium conditions. The electrolyte load on the I-X was estimated from specific resistance measurements and a published correlation* to NaCl concentrations. Based on this estimate, the total load was 59 grains NaCl, or 8.2% of the static capacity. The NaCl leakage was 1 ppm or less throughout days 18 through 48.

The AgCl column used during this test was the same one used during the three previous tests. Since it performed as required, this test demonstrated that it's useful life is greater than 138 days (120 + 73 + 120 + 4 x 48, or 505 man-days).

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*Kunin, R., Ion Exchange Resins, 2nd Ed., John Wiley & Sons, Inc., New York, 1958, p. 456.

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The RT used during this test has a useful volume of 8.4 liters. However, with this tank in the recycle loop, the total volume of the recycle loop is 10.1 liters. In addition to this volume - approximately (1400 + 785 + 450)/1.25, or 1.6 liters of recycle liquor were withdrawn for samples or lost by spillage. Therefore, the specific volume required for this test was (10.1 + 1.6)/48 x 4, or 61 milliliters/man-day. This value, of course, is less than anticipated because the average concentration of solids in the feed water was only 1.9% instead of 3%. Onboard a manned spacecraft the RT should be sized to accommodate 3% solids in the urine and urinal flush water - which means that the required RT specific volume will be 61 x 3.0/1.9 or 100 milliliters man-day.

Finally, as shown in the next sub-section, the septums used for removing sterile water samples also have a limited useful life - which means that if they are provided in a flight system, they too are expendable items. Since the septum which failed between the 4th and 7th day had been installed before the previous 30-day test, and it had been pierced at least once each day - this test demonstrated that the useful "biological" life of a septum is on the order of 30 samples.

8.2.3 Water Quality

The quality of the raw condensate samples and the treated water collected during the 48-day, high-solids test was determined by in-house analyses and selected detailed analyses at the NASA MSC. The results obtained are presented in Tables 18 through 21, and discussed in the following paragraphs.

8.2.3.1 Raw Condensate

Table 18 and 21 indicate that increasing the concentration of solids in the recycle loop did not have any significant effect on the quality of the raw condensate. The only trends apparent are (1) a small decrease in specific resistance, and (2) an increase in the ammonia concentration. However, in spite of these variations the ACF and I-X had sufficient capacity to assure the production of extremely pure potable water (see Table 20).

The day-to-day variations in the water quality parameters suggests poor reproducibility of the analyses and/or true variations during each day's test. To gain a better understanding of this problem several samples of the raw condensate and the ACF effluent were taken on the 41st and 48th day - and submitted to the Nalco Chemical Company in Chicago, Illinois for TOC analysis. The results obtained are as follows.

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Table 18 - RAW CONDENSATE QUALITY DURING HIGH SOLIDS TEST

Test Day No.	Count/100 ml Aerobic	Anaerobic	Coliform	Yeast & Mold	Sp.Res. (Kohm-cm)	pH	NH$_3$ (ppm)	COD (ppm)	TOC (ppm)
1	0	-	-	-	28	4.9	<0.2	-	-
2	-	-	-	-	31	5.0	"	-	-
3	-	-	-	-	29	4.7	"	-	-
4	55	-	-	-	22	4.1	"	71	-
5	-	-	-	-	33	4.3	"	-	-
6	-	-	-	-	43	4.7	"	-	-
7	98	-	-	-	41	4.6	"	-	40
8	-	-	-	-	36	4.7	"	-	-
9	-	-	-	-	34	4.6	"	-	-
10	102	-	-	-	28	4.3	"	-	22
11	-	-	-	-	29	3.9	"	-	32
12	96	-	-	-	23	4.0	"	-	-
13	-	-	-	-	20	4.0	"	-	-
14	92	-	-	-	20	3.9	"	-	28
15	-	-	-	-	19	-	0.5	-	-
16	-	-	-	-	19	-	<0.2	-	-
17	89	-	-	-	19	3.9	1.0	-	31
18	0	-	-	-	20	4.0	0.4	104	26
19	-	-	-	-	45	5.9	<0.2	78	27
20	-	-	-	-	37	6.0	"	104	-
21	-	-	-	-	36	5.0	0.8	150	38
22	-	-	-	-	29	5.8	"	134	-
23	-	-	-	-	24	5.9	"	-	-
24	0	-	-	-	24	5.9	"	130	37
25	-	-	-	-	25	5.8	"	108	-
26	-	-	-	-	28	5.5	1.1	124	37
27	-	-	-	-	26	5.6	1.0	74	-
28	-	-	-	-	30	4.8	1.2	92	30
29	-	-	-	-	26	5.3	0.7	-	-
30	-	-	-	-	23	5.4	<0.2	60	28
31	0	-	-	-	25	5.5	1.8	130	-
32	-	-	-	-	21	5.5	1.7	120	49
33	0	-	-	-	21	5.4	3.0	96	-
34	-	-	-	-	7	4.8	10.0	72	22
35	-	-	-	-	15	5.1	"	64	28
36	-	-	-	-	42	5.6	2.0	50	-
37	-	-	-	-	44	4.9	<0.2	58	-
38	-	-	-	-	30	5.1	"	76	-
39	-	-	-	-	19	5.2	"	94	30
40	0	-	-	-	18	5.4	0.9	84	-
41	-	-	-	-	19	4.0	-	-	34
42	0	-	-	-	19	4.1	1.2	116	30
43	-	-	-	-	18	4.1	<0.2	118	-
44	0	-	-	-	20	4.0	<0.2	114	-
45	-	-	-	-	14	4.1	0.9	196	58
46	0	-	-	-	9	4.0	6.0	184	-
47	-	-	-	-	13	4.1	10.0	138	40
48	0	-	-	-	55	4.1	-	-	27

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Table 19 - FLUSH WATER QUALITY DURING HIGH SOLIDS TEST

Test Day No.	Aerobic	Anaerobic	Coliform	Yeast & Mold	Sp.Res. (Kohm-cm)	pH	NH_3 (ppm)	COD (ppm)	TOC (ppm)
	Count/100 ml								
1	-	-	-	-	-	-	<0.2	-	-
2	-	-	-	-	-	-	"	-	-
3	-	-	-	-	-	-	"	-	-
4	0	-	-	-	-	-	"	69	-
5	-	-	-	-	44	6.8	"	-	-
6	-	-	-	-	57	6.2	"	-	-
7	0	-	-	-	-	-	"	-	40
8	-	-	-	-	-	-	"	-	-
9	-	-	-	-	-	-	"	-	-
10	0	-	-	-	-	-	"	-	27
11	-	-	-	-	-	-	"	-	24
12	0	-	-	-	31	5.2	"	-	-
13	-	-	-	-	21	5.5	"	-	-
14	0	-	-	-	17	5.4	"	-	24
15	-	-	-	-	-	-	"	-	-
16	-	-	-	-	-	-	"	-	-
17	0	-	-	-	32	5.0	0.5	72	24
18	0	-	-	-	31	6.6	0.3	22	11
19	-	-	-	-	21	6.8	<0.2	32	19
20	-	-	-	-	22	6.6	"	50	-
21	-	-	-	-	19	6.6	0.8	52	57
22	-	-	-	-	21	6.9	0.7	54	-
23	-	-	-	-	18	6.9	0.9	-	-
24	0	-	-	-	18	6.9	0.8	42	16
25	-	-	-	-	20	6.9	"	56	-
26	-	-	-	-	19	7.0	1.0	60	21
27	-	-	-	-	19	6.9	"	86	-
28	-	-	-	-	18	6.9	1.2	66	22
29	-	-	-	-	18	6.9	0.7	78	-
30	-	-	-	-	11	6.7	0.3	46	40
31	0	-	-	-	12	6.6	2.5	44	-
32	-	-	-	-	12	-	3.2	44	32
33	0	-	-	-	20	6.2	1.7	66	-
34	-	-	-	-	13	6.1	4.7	52	48
35	-	-	-	-	30	6.1	1.2	32	23
36	-	-	-	-	35	5.8	<0.2	64	-
37	-	-	-	-	32	5.6	"	76	-
38	-	-	-	-	12	5.9	"	80	-
39	-	-	-	-	13	5.9	"	66	27
40	0	-	-	-	24	5.8	"	64	-
41	-	-	-	-	24	5.7	"	72	19
42	0	-	-	-	34	5.8	1.0	56	21
43	-	-	-	-	38	5.8	1.7	60	-
44	0	-	-	-	40	5.7	1.8	58	-
45	-	-	-	-	43	5.9	1.6	50	24
46	0	-	-	-	11	5.9	9.0	110	-
47	-	-	-	-	8	6.0	10.0	86	36
48	0	-	-	-	16	6.0	6.0	84	24

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Table 20 - <u>POTABLE WATER QUALITY DURING HIGH SOLIDS TEST</u>

Test Day No.	Aero-bic	Anae-robic	Coli-form	Yeast & Mold	Sp.Res. (Kohm-cm)	pH	NH_3 (ppm)	COD (ppm)	TOC (ppm)
	Count/100 ml								
1	0	0	0	0	22	6.5	8.5	80	-
2	-	-	-	-	21	6.6	5.9	-	-
3	-	-	-	-	25	6.2	5.3	-	-
4	0	0	0	0	24	6.4	6.0	70	-
5	-	-	-	-	23	6.3	5.5	-	-
6	-	-	-	-	23	6.3	5.9	-	-
7	4	0	0	0	23	6.3	5.9	78	38
8	-	-	-	-	24	6.4	6.0	-	-
9	-	-	-	-	22	6.4	6.6	-	-
10	120	0	0	0	22	6.4	6.0	81	36
11	-	-	-	-	19	6.6	6.5	-	25
12	260	0	0	0	18	6.6	7.6	-	-
13	-	-	-	-	18	6.7	6.7	-	-
14	210	0	0	0	19	6.6	7.5	83	20
15	-	-	-	-	19	6.9	6.7	-	-
16	-	-	-	-	18	6.7	8.2	-	-
17	214	0	0	0	18	6.7	7.8	-	28
18	0	0	0	0	150	6.5	<0.2	-	14
19	-	-	-	-	650	6.6	"	-	11
20	-	-	-	-	820	6.5	"	-	-
21	-	-	-	-	480	6.6	"	-	30
22	-	-	-	-	1230	6.5	"	-	-
23	-	-	-	-	710	6.0	"	-	-
24	0	0	0	0	1125	6.3	"	-	13
25	-	-	-	-	730	6.7	"	-	-
26	-	-	-	-	740	6.5	"	-	21
27	-	-	-	-	670	7.9	"	-	-
28	-	-	-	-	1340	6.2	"	-	15
29	-	-	-	-	370	6.2	"	-	-
30	-	-	-	-	870	6.6	"	-	27
31	0	0	0	0	740	6.4	"	-	-
32	-	-	-	-	1120	6.5	"	-	30
33	0	0	0	0	560	6.2	"	-	-
34	-	-	-	-	770	6.3	"	-	22
35	-	-	-	-	540	6.3	"	-	23
36	-	-	-	-	720	6.2	"	-	-
37	-	-	-	-	560	6.5	"	-	-
38	-	-	-	-	550	5.6	"	-	-
39	-	-	-	-	980	5.6	"	-	23
40	0	0	0	0	910	6.0	"	-	-
41	-	-	-	-	910	6.1	"	-	-
42	0	0	0	0	1140	6.3	"	-	27
43	-	-	-	-	1040	6.1	"	-	-
44	0	0	0	0	900	6.0	"	-	-
45	-	-	-	-	800	5.7	"	-	26
46	0	0	0	0	840	6.1	"	-	-
47	-	-	-	-	800	6.1	"	-	32
48	0	0	0	0	480	6.6	"	-	28

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Table 21 - DETAILED WATER QUALITY DURING HIGH SOLIDS TEST

Test Day No.		pH	Sp.Res. (Kohm -cm)	NH$_3$	Cl	Cd	Cu	Fe	Mn	Ni	Ag	Zn	TOC	TDS
						ppm								
7	Condensate	4.0	28	-	-	<.01	.05	0.1	.05	0.1	.05	.08	36	4.0
	Flush	5.4	33	-	-	"	"	0.3	"	0.4	.40	.03	16	27.0
	Potable	6.1	30	-	-	"	"	0.1	"	0.1	.05	.03	22	4.0
14	Condensate	4.0	23	-	-	<.01	.05	0.1	.05	0.1	.05	.03	27	3.0
	Flush	4.9	25	-	-	"	"	0.4	"	0.5	.65	.10	21	31.0
	Potable	4.6	25	-	-	"	"	0.1	"	0.1	.05	.03	27	1.0
18	Condensate	4.2	30	0.2	7.4	-	-	0.1	.05	0.1	.05	.01	33	4.7
	Potable	5.8	275	<0.1	<0.3	-	-	"	"	"	"	"	14	7.1
21	Condensate	4.8	42	0.2	9.2	-	-	0.1	.05	0.1	.05	.01	42	22.1
	Flush	5.9	25	0.8	10.0	-	-	"	"	"	.20	"	64	36.5
	Potable	5.6	290	<0.1	<0.3	-	-	"	"	"	.05	"	34	4.2
28	Condensate	4.9	40	0.3	11.0	<0.1	.05	0.1	.05	0.1	.05	.01	27	18.9
	Flush	5.5	25	0.5	7.4	"	"	"	"	"	.35	"	23	26.9
	Potable	5.5	300	0.2	8.4	"	"	"	"	"	.05	"	20	1.0
34	Condensate	6.0	15	4.9	31.0	<.01	.05	0.1	.05	0.1	.05	.02	22	29.3
	Flush	5.9	30	1.8	17.0	"	"	"	"	"	"	.01	48	134.1
	Potable	4.8	122	<0.1	<0.3	"	"	"	"	"	"	"	22	2.4
42	Condensate	4.2	20	1.3	9.2	<0.1	.05	0.1	.05	0.1	.05	.01	35	6.2
	Flush	6.5	42	0.8	9.2	"	"	"	"	"	"	"	26	20.4
	Potable	6.3	690	<0.1	<0.3	"	"	"	"	"	"	"	36	2.0
45	Condensate	4.3	17	1.7	14.0	<0.1	.05	0.1	.05	0.1	.05	.01	58	30.8
	Flush	6.7	42	0.8	3.7	"	"	"	"	"	"	"	24	12.5
	Potable	6.1	700	<0.1	<0.3	"	"	"	"	"	"	"	26	1.0
48	Condensate	6.4	16	1.5	2.6	<0.1	.05	0.1	.05	0.1	.05	.01	27	13.2
	Flush	7.1	55	5.1	14.0	"	"	"	"	"	"	"	24	27.3
	Potable	6.6	480	0.2	<0.3	"	"	"	"	"	"	"	28	2.5

Hours After Start	TOC (ppm) Condensate #41	Condensate #48	ACF Effluent #41	ACF Effluent #48
0.5	25	32	--	--
1.0	--	--	16	20
1.5	35	--		--
2.0	--	24	17	--
2.5	--	--	--	18
3.5	41	--	--	--
4.0	--	--	26	--
5.0	--	18	--	--
5.5	35	--	--	16
6.0	--	--	17	--
7.0	--	15	--	--
7.5	--	--	--	16
8.0	--	23	--	--

From this data it must be concluded that the quality of the raw condensate does actually vary during each test. The most probable cause of this phenomena is the composition of the feed water - which varies during each test from "fresh" waste water to recycle liquor. For example, when treated urine and urinal flush water are added to the recycle loop the feed into the distillation unit is primarily these fresh wastes, with some recycle liquor; eventually, the fresh wastes are all passed through the unit and the feed is then all recycle liquor. Since fresh wastes contain more low boiling point constituents than recycle liquor, it is not surprising that the quality of the raw condensate varies during each day (test).

The biological contamination experienced in the raw condensate during days 4 through 17 is attributed to the sampling technique used. Initially, to facilitate the removal of large volumes, raw condensate samples were withdrawn through a line which did not include a BF at the outlet. At the end of the 17th day a BF was installed at this outlet and all biological samples taken after that time were withdrawn through a septum. Obviously, without a BF at the sample outlet airborne microorganisms were able to migrate into the line, where they propagated and were subsequently flushed out with samples.

8.2.3.2 Treated Condensate

The flush water (raw condensate passed through the ACF and dosed with AgCl ions) was found to contain no aerobic bacteria on each day tested. However, the potable water (see Table 20) was found to contain aerobic bacteria on days 7 through 17. At the end of the 17th day the ACF and the I-X were replaced so that both biological and chemical useful

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lives of at least 30 days could be demonstrated before the end of this test. After this change all of the potable water samples were sterile.

There are two possible causes for aerobic bacteria in the potable water samples taken on days 7 through 17. First, it could have been contamination within the I-X, which was installed at the beginning of the previous 30 day test - and chemically expended after untreated urine was processed on days 15 and 16 of that test; thus, this I-X had already demonstrated a useful biological life of at least 34 days before aerobic bacteria were discovered. The second, and the most plausible cause, is failure of the septum at the I-X outlet; upon inspection it was observed that septum was degraded as the result of being penetrated by an 18-gauge needle over a 47-day period of nearly daily sampling. However, even though the exact cause of biological contamination was not determinable, this test did demonstrate that the useful biological and chemical lives of the ACF and I-X are both at least 30 days.

Since the I-X was chemically expended during the previous test, the samples taken on days 1 through 17 of this test contained excessive amounts of ammonia - even though the ammonia content of the raw condensate and flush water were both acceptable. As explained before, this is the result of metallic ions displacing ammonia from the I-X resin. However, after the I-X was replaced at the end of the 17th day - the quality of the potable water was outstanding.

In addition to the detail analyses presented in Table 21, several samples were analyzed for barium, arsenic, mercury, lead and chromium. In all cases the concentrations were found to be less than the detection limits - which were less than the potability limits.

The presence of iron and nickel in the flush water suggests corrosion of a stainless steel component. Iron was first detected on day 14 of the previous test; on day 28 of the same test, both nickel and iron were detected. The presence of both elements persisted until the post-treatment loop change-over at the end of day 17; neither iron nor nickel were detected thereafter. The offending hardware item was probably a 400 series, stainless steel tube fitting located between the AgCl column and bladder tank. This fitting and line was replaced at the time of post-treatment change-over by a nylon fitting and translucent polyethylene tubing; this change was effected to permit visual observation of fluid flow. The impact of the corrosion episode on the hardware is difficult to assess and depends upon the category of corrosive attack which was not evident from visual examination of the suspect fitting. If the metal contamination arose from

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uniform corrosion, very little impact would be exerted on useful life because of the small quantity of metal lost.* However, if pitting corrosion occurred (not uncommon in stainless steel), even a small amount of metal loss could shorten the useful life of the component. Based on experience to date, austenitic stainless steels have not been a problem in regard to corrosion and have been the predominant construction material used in the test hardware.

8.2.4 Recalibration

On the day following the 48th test day the distillation unit was recalibrated once again to determine if it's performance had been degraded after 90 + 48, or 138 days of operation with wastes. Tap water was fed into the unit, and the excess feed (recycle) was discharged into a sewer. After two hours of operation the condenser pressure stabilized at 38 torr; the recovery rate was then found to be 1.6 kg/hr - which is sightly higher than shown by the precalibration data presented in Figure 13 (page 4-4). Thus, it is concluded that the performance of the distillation had not degraded. The slightly higher performance attained is attributed to the redistribution of grease within the compressor.

8.3 Conclusions & Recommendations

The 48-day, high-solids test achieved it's objective - namely, (1) to determine the effect of solids concentration on water quality, and (2) determine the maximum concentration which permits stable operation. The previous subsections indicate that if operated properly, the distillation unit will perform reliably with a recycle loop which contains up to 56% solids obtained from treated urine and urinal flush water. Also, the effect of these solids on the quality of the raw condensate is negligible over the range tested. However, since the recycle liquor was extremely viscous near the end of the test, and a safety factor is usually desired - the recommended safe upper limit with urine and pretreat solids is 50%.

This test also yielded a vast amount of new design and operating data - which are summarized as follows.

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*414.5 liters of water were processed through the loop during the entire test. If all of this water contained the worst case concentration of iron (2.7 mg/l) & nickel (0.8 mg/l), a total of only 1.5 grams of metal would have been lost; the actual loss is probably less than 1/2 of the worst case amount.

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A. The timing gears on the vapor compressor must be periodically lubricated - approximately once very 500 hours. Future designs should include an automatic lubricant dispenser and distributor to avoid this routine maintenance.

B. To achieve reliable operation at solids concentrations above 35%, raw condensate withdrawn from the condenser must be fed back into the evaporator during the shutdown sequence. This procedure avoids excessive solids concentrations and assures a clean evaporator when the distillation unit is restarted.

C. When operating with treated urine solids, the recovery rate attainable by the distillation unit is a function of the feed rate. To maximize recovery rate the feed rate must be maintained above 6.0 kg/hr over the entire range of 0 to 50% solids.

D. Approximately 20% of the treated urine solids precipitate in the recycle loop. To avoid plugging this loop, a recycle tank which contains a 25-micron filter should be located immediately downstream of the recycle pump.

E. The distillation rate attainable with 56% solids is slightly more than one-half the rate attainable with tap water.

F. The overall heat transfer coefficient for the distillation unit varies linearly from 120 cal/hr-cm^2-$^{\circ}$C to 30 cal/hr-cm^2-$^{\circ}$C, as the solids content of the recycle loop is varied from 0 to 56%.

G. The slip constant for the rotary-lobe compressor is 465 $\pm$ 35. Since this is almost twice the value predicted by previous investigators it is concluded that higher performance can be achieved by reducing demister, inlet and outlet pressure losses.

H. The useful life of a 25-micron filter (RF) in the recycle loop is at least 120 man-days.

I. The useful life of biological filters (BF) at the flush and potable water outlets is at least 45 days.

J. The useful life of biological filters (BF) loaded with AgCl particles is at least 60 days.

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K. The useful life of the activated carbon filters (ACF) and deionizer columns (I-X) used is at least 30 days.

L. The useful life of the AgCl column used is at least 505 man-days.

M. The specific volume required for disposing of treated urine solids, in a recycle tank, is 61 milliliters/man-day if the treated urine and urinal flush water contains 1.9% solids.

N. The septums used for withdrawing sterile water samples have a useful biological life of approximately 30 penetrations by an 18-gage needle.

C   H   E   M   T   R   I   C

TERMINATED WASH-LOOP TEST

The 30-day, wash-loop test reported in Section 6 was performed with a DuPont Model B5 Permasep unit - using Miranol C2M as the surfactant, and Hyamine 1622 as the germicide added to the permeate for washing. The Permasep unit survived the test, but the feed pressure increased continuously during the test as solids were accumulated within the unit. In addition, the total solids and total carbon content of the raw permeate exceeded 1000 ppm and 800 ppm, respectively, by the end of the test. Since the screening tests reported in Appendix A had shown that Vancide BN is more easily rejected by DuPont's B5 material than quatenary ammonium salts like Hyamine 1622, Contract NAS9-9191 was modified to include another wash-loop test using Vancide BN instead of Hyamine 1622. The following subsections describe the procedure, results and conclusions of this test.

Another objective of this test was to evaluate the performance of a rotary-lobe compressor which does not require the use of a lubricant to reduce internal clearances, and thereby achieve reasonably good performance. The following subsection describes the work performed to achieve this objective.

The compressor work indicates that coatings can be as successful as lubricants in minimizing internal clearances. However, the wash-loop test results indicate that even more work is required to demonstrate that RO can be successfully used to recycle wash water onboard manned spacecraft.

9.1 Compressor Development

The rotary-lobe compressor provided in the distillation unit is a custom design which utilizes the lobes and timing gears from a Gardner-Denver Company Model 2PDR4 compressor. For this test a duplicate compressor was fabricated. However, instead of using Texaco's Unitemp 500 lubricant to minimize internal clearances, the lobes and housings were coated with DuPont's Buff Sealer.

The coating was applied in successive layers; after each layer had dried the compressor was operated at full speed in ambient air on a bench fixture to "rub off" the high spots. The effective clearance remaning after each application of buff sealer was measured by plugging the compressor inlet port with a vacuum gage to read the head rise with no flow. With zero flow through the compressor, productive speed was zero and slip speed was the actual speed. Slip constants corresponding to each pressure rise, therefore, were directly calculated. When a slip constant equal to that achieved by the first compressor was reached, the effective clearance of the two machines was equal, and applications of buff sealer were terminated.

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The second compressor was installed in the distillation unit and calibrated with tap water. The results obtained were essentially the same as for the first compressor (see Figure 13). Therefore, the wash-loop test was initiated without any additional work on the second compressor.

9.2 Test Set-Up and Procedure

This test was performed using essentially the same equipment and arrangement provided for the 30-day, wash-loop test reported in Section 6. The quantity and the composition of the wash solutions and the commode flush water were varied - in addition to the test procedure.

Before this test was started, the NASA MSC revised the anticipated quantity and composition of the wash water required onboard a modular space station. To conform with these requirements, the following quantities and additives were used for each man-day of testing.

	Showers	Clothes	Dishes	Experiments
Water (nom.)	5.5 1	16.8 1	6.8 1	0.75 1
Miranol C2M*	5.5 gm	16.8 gm	6.8 gm	5.0 gm
Vancide BN**	0.0	17.0 gm	6.8 gm	0.75 gm

The shower water and clothes wash water were used for the purposes intended; experiment water was simply recycled because of the wide variety of contaminants possible. Dish water was contaminated with 31 gm/man-day of roast beef hash to simulate the protein, carbohydrate and fat composition of typical food residue. Evaporation losses were compensated for each day by "topping-off" the used wash water each day to 29.5 liters/man.

Commode flush water was used at the rate of 1.5 liters/man-day. The quantity of PS used was reduced from 30 gm/man-day, as used during the 30-day, wash-loop test, to 13.5 gm/man-day - so that the quantity of PS used in the wash loop would be the same as determined necessary for the urine loop by the experiments reported in Section 7.

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* Solids in 15% solution
**Powder

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At the beginning of this test the RO module was fed used wash water at a two-man rate, and the brine bleed-off rate was adjusted to achieve a water yield near 95%. However, when the water permeability of the module began to decrease, the bleed rate was increased to avoid feed pressures higher than 600 psig. Also, part of the brine was recycled to keep the RO running time about the same as the distillation time. Eventually, the RO feed rate was reduced to the one-man rate to avoid running times longer than one shift (8 hours).

One modification made to the distillation unit, besides the new compressor, was changing a bowl drive gear to increase the nominal bowl speed from 205 rpm to 224 rpm. This change was made to increase the head on the suction side of the recycle pump, by a factor of $(224/205)^2$, and thereby possibly improve the useful life of the tubing in the pump.

During the first six days of the test the permeate was only dosed with AgCl ions before reuse. After that, an ACF was added to improve water quality.

9.3 Results & Discussion

The wash-loop was operated for 12 days, plus one day for recalibration. The test was terminated at the end of the 12th day because the performance of the Permasep unit had degraded to the level where further testing would not have yielded any meaningful data.

During this test the distillation unit experienced one failure - namely, on the 4th day the roll-pin in the motor-compressor coupling failed as the result of excessive wear. A new roll-pin was installed and the test was resumed. Since this pin had also failed on the 23rd day of the previous test series - this test re-emphasized the previously made conclusion that the coupling should be redesigned.

Unscheduled, but anticipated, maintenance was limited to changing the 3-micron prefilter, located upstream of the RO feed pump, during the 6th day. On days 7, 8 and 9 the RO unit did not process real wash water - instead, it was operated with deionized tap water to see if the Permasep unit could be "cleaned".

9.3.1 Thermodynamic Performance

The daily inputs, outputs and running times are presented in Table 22. The anomalies and trends in this data are explained as follows.

Table 22 - PERFORMANCE SUMMARY FOR TERMINATED WASH-LOOP TEST

Test Day No.	RO Feed Pressure (psig)	Permeate Produced (gm)	RO Run Time (hrs:min)	CD Run Time (hrs:min)	Condensate Produced (gm)	Recycle Dumped (gm)	Purge Losses (gm)
1	490 - 525	51100	6:12	8:00	9355	0	168
2	520 - 610	48066	6:18	8:00	11505	0	172
3	600	48935	6:52	9:00	10831	0	120
4	600	45145	7:12	10:30	14735	0	160
5	600	50370	8:42	11:25	12240	130	255
6	600	49145	9:37	10:15	10290	0	171
7 & 8	RO Processed Miranol Solution		-	Distillation Unit Not Operated			
9	RO Processed Deionized Tap Water		-	Distillation Unit Not Operated			
10	600	26770	6:03	5:35	3940	400	161
11	600	25250	6:20	7:00	6200	200	130
12	600	26430	6:05	6:40	6665	0	122

A. Initially, the feed pressure was approximately 500 psig - as the precalibration data indicated it should be with a water permeation rate near 135 ml/minute (see Figure 18). However, during the second day an increasing feed pressure became apparent. The brine bleed rate was then increased to limit the feed pressure to 600 psig - the maximum pressure recommended by DuPont for the B5 Permasep module.

B. The water permeability continued to decrease from days 1 through 6, as illustrated by Figure 26. However, after processing clean water for three days the water permeability leveled off so that the water permeation rate was about one-half the feed rate for the remainder of the test. From this result it is concluded that the B5 Permasep unit cannot achieve a reasonably long useful life when processing 3μ prefiltered wash water unless the yield is less than 50%.

C. Accurate mass balance and yield determinations could not be made during this test - because when the Permasep module was "cleaned" during days 7, 8 and 9 the brine was discarded, to avoid excessive solids loading during days 10, 11 and 12.

9.3.2 Expendables Required

This test was too short to demonstrate that the useful lives of the Recycle Filter (RF), Biological Filters (BF), and the Activated Carbon Filter (ACF) are greater than 30 days - as had been done during the High Solids Test. However, it did demonstrate that:

A. The reduced quantity of PS used in the commode flush water is capable of maintaining the recycle liquor in a sterile condition.

B. The useful life of the AgCl column, which had been used since the beginning of this program, is greater than 505 + 2 x 6 + 1 x 3, or 520 man-days.

C. The useful life of the $3\text{-}\mu$ RO prefilter is less than 2 x 6, or 12 man-days.

The useful life of the RO prefilters, of course, is a function of the pressure drop provided by the pump(s) upstream of the filters. Figure 27 shows that during the first 6 man-days of operation the pressure drop across the 3μ filter was always in the range of 1.3 to 1.8 psi. However, during the 4th day (man-days

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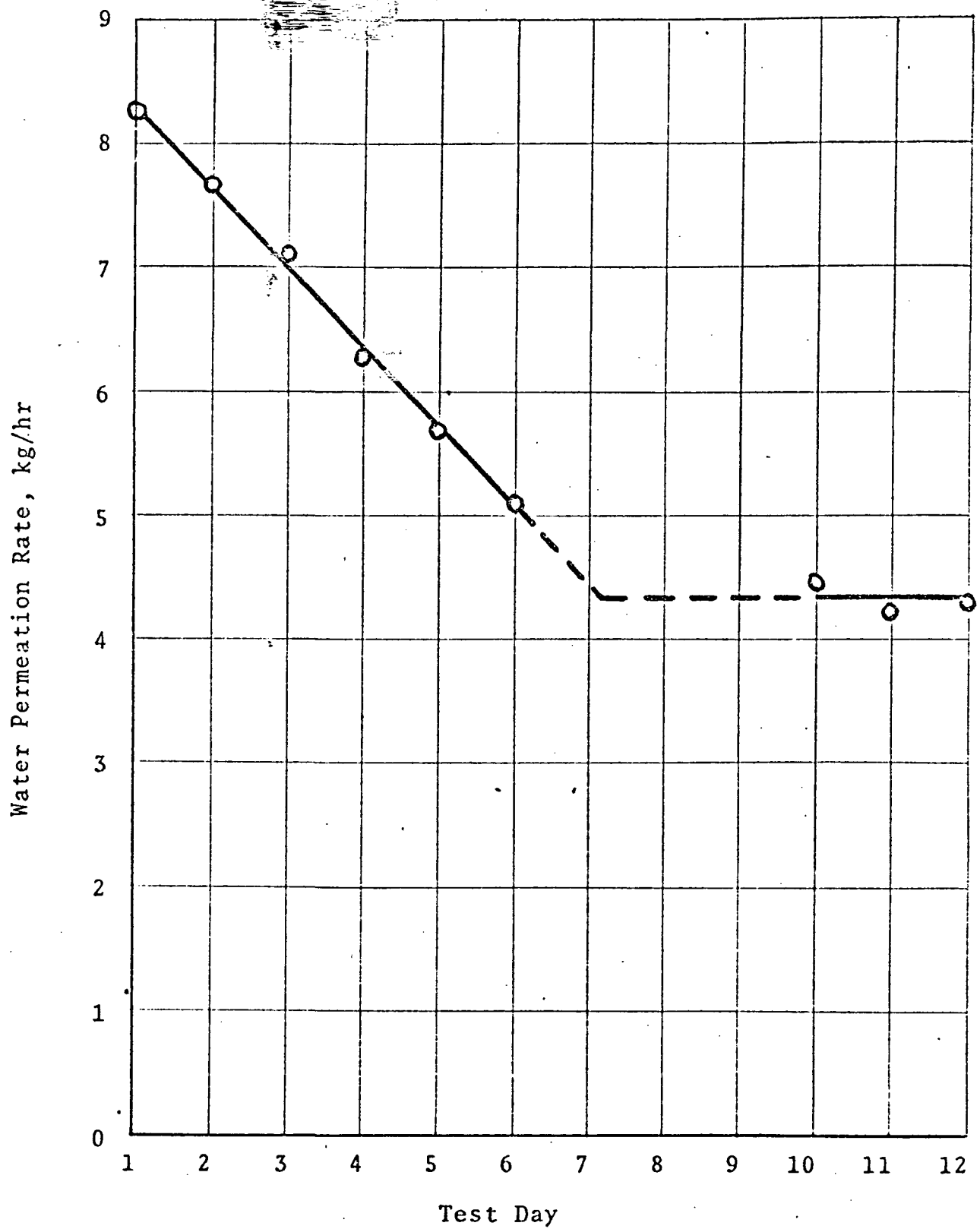
Figure 26　　FOULING OF MODEL B5 PERMASEP MODULE

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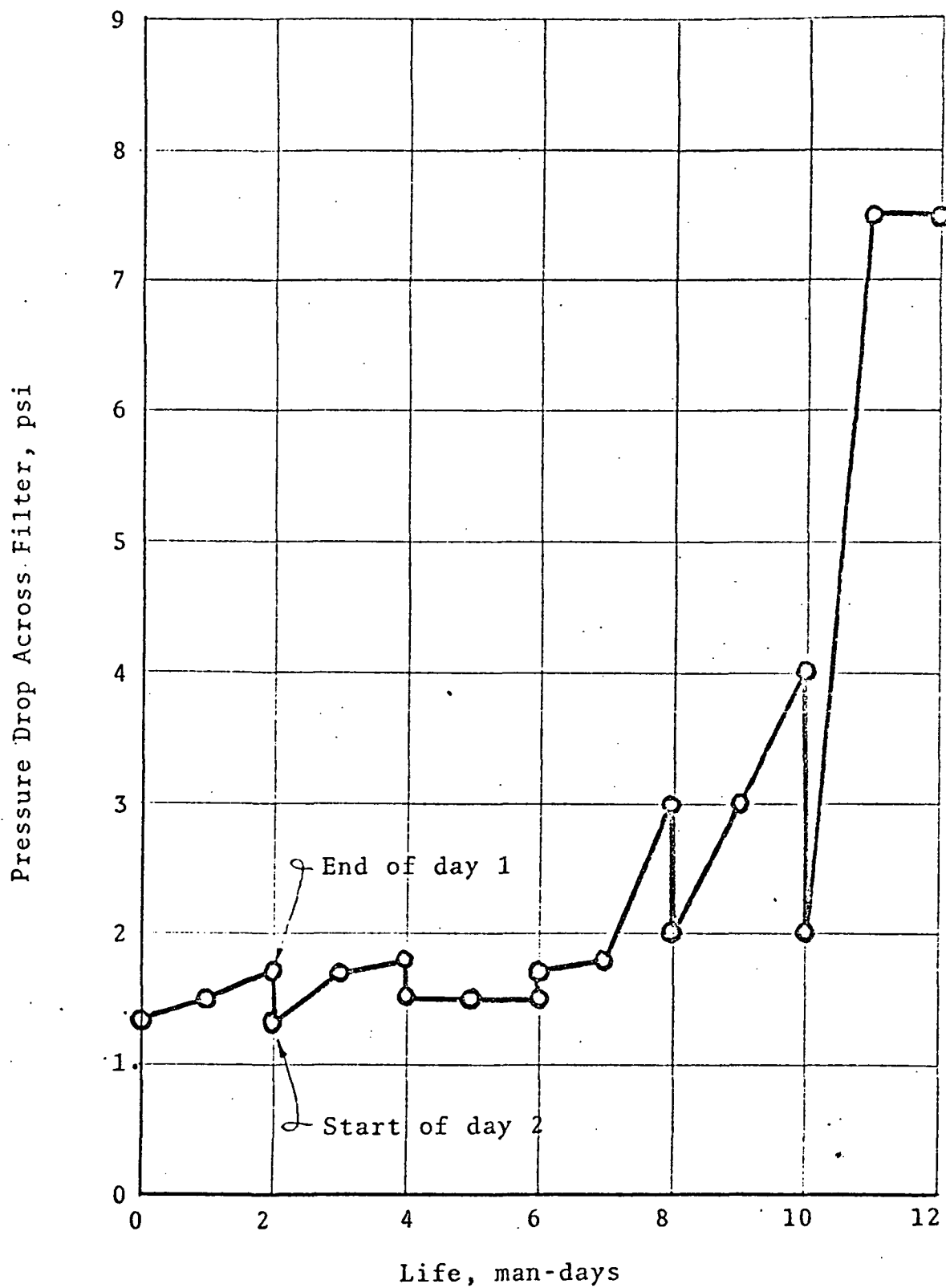
Figure 27   PLUGGING OF 3-MICRON REVERSE OSMOSIS PREFILTER

C   H   E   M   T   R   I   C

7 & 8) it increased to 3.0 psi, and from there on was never less than 2.0 psi. From this result it is concluded that the "practical" useful life of this filter is only 6 man-days.

The first nine days of this test were performed without charcoal filtration of the permeate. At the end of the 9th day three ACF's, each containing 160 grams of activated carbon, were installed in series downstream of the AgCl column.

Septums were located at the inlet and outlet of each ACF for sampling purposes. The total COD of the raw permeate filtered during the 10th day was 14.25 grams - as computed from the average flow rate and COD analysis of several samples. By the end of the 10th day the COD of the effluent from the first ACF had risen from near zero to 100 ± 5 ppm. The total COD adsorbed, therefore, was 11.73 grams - which corresponds to a loading factor of 11.73/160, or 0.073 gm COD/gm charcoal. On the next day the first column adsorbed another 4.76 grams of COD, but the effluent COD was up to 126 ppm. On the third day (day 12) another 6.0 grams of COD was adsorbed, and the effluent COD was up to 156 ppm. Thus, the cumulative loading factor for the first ACF was (11.73 + 4.76 + 6.00)/160, or 0.140 gm COD/gm charcoal.

The second and third ACF units (connected in series to the first unit) exerted almost no impact in lowering the COD of the effluent from the first ACF unit through days 10, 11 and most of day 12. When the effluent from ACF #1 reached 156 ppm COD in the last hours of day 12, the two ACF units, taken together, lowered the COD by only 30 ppm. From the standpoint of COD adsorbed, the first ACF adsorbed 80 to 60% of the incident COD while the two ACF units downstream, taken together, adsorbed almost none of the incident COD.

The above performance may be due to either concentration effects (the COD of the influent of the first unit was 2 to 4 times higher than the influent of the 2nd and 3rd units) or composition; that is, the permeate contained some organic compounds which were readily adsorbed as well as some compounds which are not readily adsorbed. Mixed composition appears to be the most plausible for the following reasons:

1. The residence time provided by ACF units 2 and 3 taken together was twice that of ACF #1; this doubling of residence time should have produced a significant depression in COD despite the 3 to 4 fold lower COD concentration.

2. Low molecular weight, highly water soluble organic species are present in waste water and are poorly rejected by most RO membranes; these same compounds are also poorly adsorbed out of water by activated carbon.

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9.3.3 Water Quality

The analyses performed during this test were limited primarily to determining the carbon content of the RO process streams. Spot checks were also made on the flush water (filtered and AgCl dosed condensate) to determine that it's pH ranged from 4.4 to 5.1, and it had a specific resistance ranging from 20 to 32 kohms-centimeter. The flush water and the recycle liquor were both found to be sterile on days 5 and 12.

Samples of the input and output streams of the RO module were analyzed by the NASA MSC for total organic carbon (TOC) and inorganic carbon (IC). Table 23 lists the average values obtained from several samples taken at regular intervals during each test day. Comparison of this data to that presented in Table 9 (page 6-18) for the 30-day, wash-loop test indicates that permeate quality is substantially better when Vancide BN is used instead of Hyamine 1622 - as predicted by the screening tests reported in Appendix A.

An interesting result of the carbon analyses was the daily variation of the carbon in the permeate - i.e., samples taken during the first hour of each daily test had carbon contents similar to the brine. This indicates that over-night, solids permeate the hollow fibers to equalize the concentration at the inner and outer surfaces. Thus, to maximize the average quality of the permeate and/or reduce the amount of charcoal for post-treatment - a flight-qualified RO subsystem should reprocess the permeate initially produced after extensive shut-downs.

9.3.4 Recalibration & Inspection

On the day following the 12th test day the distillation unit was recalibrated with tap water. Recovery rates of 1.18 and 1.2 kg/hour were obtained with condenser pressures of 36 and 39 torr, respectively. Comparison of these data points to those presented in Figure 13 (page 4-4) indicates that the performance had degraded.

The distillation unit was then disassembled and inspected. As usual, the evaporator was found to be clean; however, some of the coating on the internal surfaces of the compressor had "flaked-off". Thus, the reduced recovery rate experienced was due to increased slippage - i.e., the volumetric efficiency of the compressor had decreased due to increased back-flow at the locations where the coating had "flaked-off".

On close inspection it was discovered that the coating had not flaked-off the cast-steel lobes; coating was only lost from the ground stainless steel surfaces which formed the end plates and the housing for the lobes. Thus, it is concluded that the

Table 23 CARBON CONTENT OF RO PROCESS STREAMS

Test Day No.	Filtered Feed		Brine		Permeate	
	TOC*	IC**	TOC	IC	TOC	IC
1	--	--	--	--	--	--
2	636	56	1450	78	210	27
3	730	59	1600	78	272	30
4	--	--	2233	84	187	45
5	--	--	3540	98	254	53
6	1600	91	3030	133	255	51
7	--	--	--	--	--	--
8	--	--	--	--	--	--
9	--	--	--	--	--	--
10	2250	125	1730	123	170	40
11	1080	122	1700	130	116	43
12	1240	92	1400	108	176	52

* TOC = total organic carbon, ppm

** IC = inorganic carbon, ppm

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coating failure was due to poor adhesion instead of any chemical or vapor attack - and that the coating would not have been lost if the stainless steel surfaces had been sand blasted or chemically etched prior to the application of the coating.

9.4 Conclusions & Recommendations

This test demonstrated that the raw permeate obtained from a DuPont B5 Permasep module processing used wash water is of substantially higher quality if Vancide BN is used as the germicide added to clothes and dish water, instead of Hyamine 1622. It also demonstrated that volumetric efficiency of a "coated" compressor can be equal to that obtained when a lubricant is used to reduce internal clearances. However, this test failed to demonstrate that the Permasep module and a coated compressor have the useful life desired for manned spacecraft.

The basic problem with the B5-Permasep module appears to be surface fouling and/or channeling. Even though the used wash water was passed through a 3μ , in-depth filter which has to be changed every 6 man-days, the module's water permeability is decreased until the water yield is 50% or less. Since this is an impractical performance level for a spacecraft, and finer pre-filter would have to be replaced more frequently than once every 6 man-days, it is concluded that other types of RO modules should be investigated.

The compressor coating problem can be solved by "roughening" the lobes and end plates before the coating is applied; additional work on this problem is not recommended.

FINAL WASH-LOOP TEST

The wash-loop tests reported in Sections 6 and 9 were performed with a DuPont Model B5 Permasep unit. The performance of this unit declined to unsatisfactory levels; surface fouling and accumulation of particulates ≤ 3 microns were the causes of performance degradation. Moreover, DuPont stopped production of the Model B5 Permasep unit and replaced it with the Model B-9. It was therefore necessary to do additional testing and evaluation to support the design of the SSP WWMS.

Contract NAS 9-9191 was further modified to perform another 30-day flush/wash water loop test. Two units, namely, DuPont's B-9 Permasep Permeator and Westinghouse's 4 x 4 Basic Module, were preselected for evaluation by NASA. Prefatory tests were to be conducted also to (1) determine the distribution of suspended solids resulting from the use of various cleansing agents, (2) establish compatability of the reverse osmosis units with the various preselected cleansing agents, and (3) select the unit for the 30-day loop test. The following subsections describe the procedure, results and conclusions of these tests.

The distillation unit's compressor described in Section 9 was modified for the flush/wash water loop test. Rotor clearances were reduced axially by machining the housing, and radially by application of buff sealer to the rotors only. Previous testing showed that buff sealer did not adhere well to stainless steel, but adherence to the cast steel rotor was better. Another objective, therefore, was to evaluate the performance of a "tight" dry compressor.

10.1 Prefatory Tests

A detailed summary of the experimental work performed is presented in Appendix C. The following conclusions are based upon the results of the experimental investigations conducted.

1. Liquid Vancide BN is the most promising germicide.

2. On the basis of filtration and miscibility, Miranol C2M is the best amphotheric detergent for the application.

3. Bar soaps (e.g., Ivory and Neutrogena) contain suspended solids which readily plug filters. Their use would also develop problems in the loop's distillation units because of solubility limits.

4. Neutrogena Rainbath Body Soap Gel is the preferred skin cleanser because the formulation does not contain any suspended solids.

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5. One hundred (100) millimicron filtration of used (soiled) wash water is required to achieve a turbidity of 1 JTU (Jackson Turbidity Unit). Turbidities of 0.1 JTU will never be practically achieved with membrane filtration; adjunct pretreatment will probably be required.

6. Both DuPont's B-9 Permasep unit and Westinghouse's 4 x 4 Basic Module are compatible with the selected detergents and germicides; namely Miranol C2M-SF, Neutrogena Rainbath Body Soap Gel, and liquid Vancide BN. The units showed no degradation in performance after 30 days of combination static storage/operational tests.

The Westinghouse unit was recommended for the flush/wash water loop test. The tubular membrane design configuration is capable of minimizing clogging and fouling, and reportedly the membrane surface can be readily cleaned using periodic soak-flush methods. It is further recommended, that all cleansing agents be prefiltered before onboard storage, so that inflight filtration requirements are reduced.

10.2 Compressor Modification

Operation of the distillation unit during the final wash-loop test provided an opportunity to operate a dry compressor with very small clearances around the dynamic elements. Conventional compressors are built with clearances on the order of 0.0100 inches, but for this test axial clearance between the rotors and housing was reduced to 0.0015 to 0.0020 inches by machining the compressor case and reworking some bearing housings. This clearance is very near the practical limit considering the accumulation of several tolerances (i.e., linear, perpendicularity, concentricity, dynamic distorsions and surface waves) added to the already-present radial and axial clearances within the ball bearings. It would have been advantageous to machine new rotors to hold the same close clearance between the rotating lobes as in the case of the housing-rotor interface, but lack of resources (time and funds) prevented this approach. Instead, the original rotors were coated with several applications of buff sealer and then operated in the compressor housing on a bench fixture to "wear off" the high spots.

After the run-in period the newly assembled compressor drew 17-1/2 inches Hg vacuum at the plugged inlet when driven at 3400 rpm on the bench fixture with ambient discharge. When the test was completed, the compressor was operated in the bench fixture again under the same conditions; it produced the same 17-1/2 inch vacuum.

10.3 RO Processor

Westinghouse's 4 x 4 Basic Module, Serial No. 4-41 was selected for the flush/wash water loop test. This unit had processed a cleansing agent test solution without pH adjustment during the prefatory tests (i.e., compatibility tests). Recalibration runs on the unit showed permeate flow rates which coincided with the original calibration.

Figures 28 and 29 show a photograph and a cut-away view of the Westinghouse unit. The module is 52" long overall and weighs 80 pounds. The tubular membranes are produced by drop-casting a special cellulose acetate base solution in parallel holes cored through a length of cylindrical resin - bonded sand matrix. The unit contains eighteen (18) tubular membranes of approximately one-half inch diameter. Water under pressure permeates through the membrane wall, then passes through the resin-bonded sand structure and is removed through side taps. A 304 stainless steel jacket 4 inches in diameter and 4 feet long supports the sand structure and end flanges. End caps or seals are joined to the 6 inch diameter flanges by bolts and are used to connect the tubular membrane elements in series, series-parallel and/or parallel hydraulic configurations.

Westinghouse Unit No. 4-41 contains 9.25 square feet of membrane area. The unit was tested by Westinghouse on August 25, 1971; the following performance characteristics were reported.

Temperature:	78°F
Pressure:	320 psi
Flow:	150 gph, 400 μmho NaCl solution
Product:	8.3 to 10.7 gal/ft^2/day (GSFD)
Rejection:	95.5 to 96.5%

10.4 Test Set-Up and Procedure

This test was performed with essentially the same equipment and arrangement used for the 30-day and the Terminated Wash-Loop tests reported in Sections 6 and 9, except the RO Processor. The following quantities of water and additives were used for each man-day of testing.

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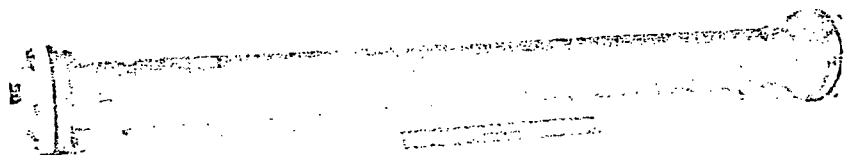
Figure 28   PHOTOGRAPH OF WESTINGHOUSE MODULE
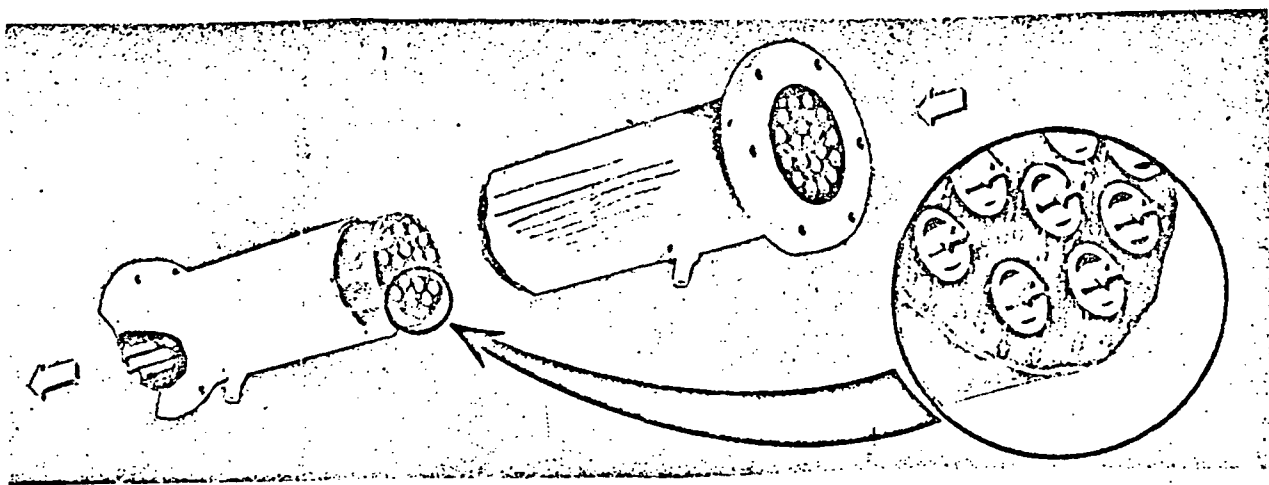
Figure 29   CUT-AWAY VIEW OF WESTINGHOUSE MODULE

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Washing Application	Quantity of Water liters	Quantity of Additive, grams		
		Neutrogena Rainbath*	Miranol C2M-SF**	Liquid Vancide BN***
Showering	3.64	11.1	0.0	0.0
Hands & Face	1.82	5.5	0.0	0.0
Clothes	16.7	0.0	20.8	16.6
Dish	6.8	0.0	8.4	6.7
Experiment	0.4	0.0	1.9	0.9

* Active ingredient 32.7%
** Active ingredient 40.0%
***Active ingredient 50.0%

The shower water, face and hands wash water, and the clothes wash water were used for the purposes intended. Experiment water was simply recycled because of the wide variety of contaminants possible. Dish water was contaminated with 31 gm/man-day of roast beef hash to simulate the protein, carbohydrate and fat composition of typical food residue. Evaporation losses were compensated for each day by addition of make-up (deionized tap) water to the used wash water so that 29.5 liters/man were available for the RO processor.

Commode flush water was simulated at the quantity of 1.5 liters/man-day. Deionized tap water plus 13.5 gm/man-day of pretreatment chemicals served as fecal flush water.

The RO unit was fed used (soiled) wash water at a two-man rate throughout the test. Initially the brine bleed-off rate was adjusted to obtain a water yield near 95%. When the water permeability of the unit began to decrease, the bleed rate was increased to avoid operating pressures higher than 450 psig. A gas-domed back-pressure regulator was installed to control the operating pressure at 425 psig. A heating tape was also wrapped around the unit to increase its operating temperature. A portion of the brine was then recycled to maintain the RO operating time about the same as the distillation units operating time. Additional flush water (deionized tap only) was processed the first two calendar test days to "run-in" the modified compressor and to obtain additional performance information.

When the distillation units drive motor failed, the flush/ wash water loop test was terminated. However, RO testing was continued. The RO unit was calibrated, flushed-out with permeate and tap water, recalibrated, and then restored to processing used (soiled) wash water again. After processing wastes for an additional eight days, the RO processing of wastes was concluded

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on the 30th calendar test day. The RO unit was then calibrated, flushed-out with tap water, and recalibrated. During this period of operation, deionized tap water was added to the system to simulate reprocessed (i.e., distilled) brine.

The permeate was not post-treated during the first ten days of testing. After that the permeate was only dosed with AgCl before reuse.

Samples of the RO unfiltered feed, RO filtered feed, permeate, brine, and the distillation unit's condensate were withdrawn each day to determine pH, turbidity, COD, specific resistivity and total solids. Samples of the distillation units recycle liquor were withdrawn each day after the 3rd test day to determine specific gravity and total solids. Occasionally sterility and MPN determinations were performed on the RO process streams by innoculating thioglycolate broth; the tubes were incubated at 35°C and inspected at 24 and 48 hours for turbidity.

10.5 Results and Discussion

The RO unit was operated for 36 days; waste water was processed for 26 days and 10 days were devoted to calibrations, flush-outs and recalibrations. The flush/wash water loop was operated for 18 days and was terminated when the distillation unit's drive motor failed.

Electric motor failure on test day #18 was attributed to carbon tracking; i.e., the insulation had degraded and caused electrical short circuiting which lead to a winding burn-out. This motor had a rated service life of 500 hours; however, it already had been in service for over 2000 hours when failure occurred. Spare motors had been on order since the begining of the contract modification but were not received in time to continue the loop tests.

On test day #13 while attempting to measure the pressure drop across the demister, manometer fluid was accidently drawn into compressor causing it to bind and stop. This action probably damaged or loosened the rotor coating (buff sealer), but the test was continued after the compressor was forced free by rotating it manually.

Unplanned maintenance required during the test was as follows.
1. The inlet to the ACF became plugged on day #13 by corrosion products from the upstream Pall cartridge filter. Both units were back-washed with deionized water and returned to service.

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2. The silicone tubing in the distillation unit's feed pump was replaced on day #16.

3. The silicone tubing in the distillation unit's condensate pump was replaced on day #17.

4. Clean orifice of micrometer regulating valve (RO bleed valve) on days #3, 6, and 7.

5. Clean and unclog RO fore pump on days #16, 18 and 30.

Unscheduled but anticipated maintenance was limited to changing the 3-micron filter located upstream of the RO feed pump on days #7, 15, 16, 18, 19, 25, 27 and 30. On days #19, 20, 21 and 22 the RO unit was calibrated, flushed-out and re-calibrated.

10.5.1 Thermodynamic Performance

Tables 24, 25 and 26, and Figures 30 and 31 summarize daily characteristics, inputs, and outputs for the RO Processor and the Distillation Unit, during the additional flush/wash water loop test. The results, trends, and anomalies in the data are described as follows.

1. During the wash-loop test (days #1 to 18 inclusive) the RO Processor yield was (100 x 927,414/927,414 + 188,799-100,893) or 91.2%. The average permeate flow rate was 927,414/124.0 or 7,480 gm/hr (16.5 lb/hr) but ranged from 9,080 g/hr (high) to 4,770 gm/hr (low). The permeate flow rate began to decline on day 13 and progressively deteriorated to the lowest figure observed on day 18 (see Figure 30); previously the permeate flow rate would decline and then recover. Solids balances presented in Table 25 indicate that accumulation of solids, approximately 400 grams (1421.9 - 1021.9) was the cause of declining permeate flow rate.

2. During the RO waste tests (days #23 to 30 inclusive) the RO Processor yield was (100 x 345,690/345,690 + 105,643) or 76.7%. The average permeate flow rate was 345,690/50.25 or 6,880 gm/hr (15.2 lb/hr), but ranged from 7,390 gm/hr (high) to 6,400 gm/hr (low). Although the permeate flow rate declined from day 23 to 30, the deterioration was not as sharp as in the wash-loop test when the yield was higher (see Figure 30). The increased flow of brine maintained "near" steady-state operations. Solids balances presented in Table 25 indicate that quantity of solids accumulated in the RO unit in this series of tests was relatively small, i.e., approximately 46 grams (596.1 - 549.9).

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Table 24   RO MODULE PERFORMANCE DURING FINAL WASH-LOOP TEST

Test Day No.	RO Permeate Produced gm	RO Brine Produced gm	Average RO Operating Pressure psig	Brine Reprocessed by RO gm	Daily RO Yield %	Make-Up Water Added* gm	Operating Time (hr:min)	Average Daily Permeate Flow Rate gm/hr
1	50430	2939	307	--	94.4	6600	5:42	8830
2	54525	2068	348	--	96.3	6300	6:00	9080
3	53225	3579	316	--	93.7	3350	6:15	8530
4	54560	3465	315	--	94.1	3170	6:18	8670
5	54245	3324	312	--	94.2	3900	6:15	8680
6	54670	3027	341	--	94.8	3300	6:15	8750
7	53420	8508	421	3508	91.3	3150	6:39	8040
8	44975	12185	425	--	78.5	2800	6:15	7190
9	49505	7365	425	--	87.4	4100	6:18	7860
10	54530	3523	400	--	94.1	3500	6:09	8870
11	52935	4575	403	--	92.0	3250	6:12	8520
12	53085	3730	405	--	93.8	3400	6:12	8560
13	51465	6118	425	--	89.4	2500	6:18	8160
14	49850	7510	425	--	87.1	3300	6:24	7760
15	48655	21198	425	14698	88.3	4000	8:33	5690
16	48985	28820	425	22822	89.1	1650	8:27	5790
17	49329	24735	425	21735	90.6	2900	9:30	5190
18	49025	42130	425	38130	92.5	4100	10:18	4770
	927,414	188,799		100,893		65,270	124:00	

Table 24 (Continued)

Test Day No.	RO Permeate Produced gm	RO Brine Produced gm	Average RO Operating Pressure psig	Brine Reprocessed by RO gm	Daily RO Yield %	Make-Up Water Added* gm	Operating Time (hr:min)	Average Daily Permeate Flow Rate gm/hr
19	Calibration							
20	Tap Water-Flush Out							
21	Tap Water-Flush Out							
22	Recalibration							
23	45080	11501	378	--	79.9	2900**	6:21	7100
24	46110	10707	425	--	81.2	16100	6:15	7390
25	44775	10618	425	--	80.8	15600	6:27	6940
26	44930	12133	425	--	79.0	16000	6:24	7010
27	42850	12710	425	--	77.0	17000	6:21	6740
28	39175	16879	425	--	69.9	15000	6:06	6400
29	42090	15170	425	--	73.6	22300	6:06	6890
30	40680	15925	425	--	71.8	19000	6:15	6490
	345,690	105,643				123,900	50:15	
31	Calibration							
32	Tap Water Flush-Out							
33	Recalibration							
34	Tap Water Flush-Out							
35	Tap Water Flush-Out							
36	Recalibration							

* Compenation for losses (evaporation, spillage and process stream sampling)
**Simulatation of distilled brine and compensation for losses.

Table 25   SOLIDS BALANCE ON RO PROCESSOR

Test Day No.	Total Solids Input			Total Solids Output					Permeate + Brine Cums.
	Feed gms/day			Brine gms/day			Permeate(recycled)gms/day		
	Vol(1)xConc(g/1)		Cum.	Vol(1)xConc(g/1)		Cum.	Vol(1)xConc(g/1)		Cum.	
1	53.369x1.08=	57.6	57.6	2.939x 8.84=26.0		26.0	50.430x0.046=2.32	2.32	28.32
2	56.593x1.13=	63.8	121.4	2.068x28.90=59.7		85.7	54.525x0.046=2.18	4.50	90.20
3	56.804x1.11=	63.0	184.4	3.579x14.89=53.3		139.0	53.225x0.060=3.19	7.69	146.69
4	58.025x1.25=	72.5	256.9	3.465x14.30=49.6		188.6	54.560x0.100=5.46	13.15	201.75
5	57.569x1.29=	74.2	331.1	3.324x18.40=61.2		249.8	54.245x0.070=3.80	16.95	266.75
6	57.697x1.31=	75.6	406.7	3.027x19.02=57.6		307.4	54.670x0.120=6.57	23.52	330.92
7	58.420x1.27=	74.1	480.8	5.000x 9.98=49.8		357.2	53.420x0.050=2.67	26.29	383.49
8	57.160x1.49=	85.1	565.9	12.185x 7.07=86.0		443.2	44.975x0.060=2.79	29.08	472.28
9	56.870x1.26=	71.6	637.5	7.365x 9.17=67.5		510.7	49.505x0.050=2.47	31.55	542.25
10	58.053x1.32=	76.6	714.1	3.523x15.66=55.0		565.7	54.530x0.140=7.64	39.19	604.89
11	57.510x1.42=	81.6	795.7	4.575x12.85=58.4		624.1	52.935x0.100=5.29	44.48	668.58
12	56.815x1.44=	81.8	877.5	3.730x14.63=54.6		678.7	53.085x0.100=5.30	49.78	728.48
13	57.578x1.36=	78.3	955.8	6.118x10.80=66.0		744.7	51.465x0.160=8.22	58.00	802.70
14	57.360x1.12=	64.2	1020.0	7.510x 9.15=68.6		813.3	49.850x0.190=9.46	67.46	880.76
15	55.155x1.50=	82.8	1102.8	6.500x 6.40=41.7		855.0	48.655x0.200=9.71	71.17	926.17
16	54.985x1.90=	104.4	1207.2	6.000x 4.59=27.6		882.6	48.985x0.150=7.36	84.53	967.13
17	54.329x2.12=	115.1	1322.3	5.000x 5.29=26.4		909.0	49.329x0.150=7.39	91.92	1000.92
18	53.025x1.88=	99.6	1421.9	4.000x 4.14=16.6		925.6	49.025x0.090=4.42	96.34	1021.94
	1015.320			87.906			927.414		
23	56.581x1.57=	89.1	89.1	11.501x5.35=61.5		61.5	45.080x0.125=5.64	5.64	67.14
24	56.817x1.17=	66.4	155.5	10.707x5.46=58.4		119.9	46.110x0.030=1.38	7.02	126.92
25	55.393x1.09=	60.2	215.7	10.618x5.82=61.9		181.8	44.775x0.035=1.56	8.58	190.38
26	57.063x1.23=	70.1	285.8	12.133x5.23=63.4		245.2	44.930x0.110=4.94	13.52	258.72
27	55.560x1.38=	76.5	362.3	12.710x5.40=68.5		313.7	42.850x0.055=2.35	15.87	329.57
28	56.054x1.55=	86.9	449.2	16.879x4.89=82.4		396.1	39.175x0.090=3.52	19.39	415.47
29	57.260x1.22=	69.9	519.1	15.170x4.35=65.9		462.0	42.090x0.060=2.52	21.91	483.91
30	56.605x1.36=	77.0	596.1	15.925x4.07=64.6		526.6	40.680x0.035=1.42	23.33	549.93
	451.333			105.643			345.690		

Table 26 DISTILLATION UNIT PERFORMANCE SUMMARY

Test Day No.	Input to Still gm	Flush Water Produced gm	Flush Water Recycled gm	Still Purge Losses gm	Recycle Liquor Dumped gm	Operating Time (hrs:mins)	Daily Average Distillation Rate gm/hr	Daily Average Specific Energy watt-hr/kg
1	14375	12340	3000	150	---	8:48	1640	54.8
2	11000	10725	3000	70	---	7:57	1510	61.3
3	6000	5990	3000	52	64	4:30	1530	60.3
4	6020	5765	3000	60	62	4:42	1397	63.8
5	5880	5685	3000	78	62	4:48	1431	63.6
6	5488	5403	3000	60	127	5:30	1495	66.9
7	7500	7050	3000	110	64	6:30	1172	84.3
8	14765	14142	3000	210	185	13:06	1131	86.7
9	10120	9315	3000	210	181	9:00	1181	81.3
10	6055	5372	3000	180	205	7:18	1150	81.7
11	7130	6590	3000	150	192	7:12	1057	90.8
12	6340	5979	3000	137	192	6:12	1100	85.7
13	8365	7990	3000	160	183	8:36	1023	102.3
14	6500	6057	3000	120	185	7:00	970	112.2
15	10000	10237	3000	250	199	10:54	1023	95.6
16	9000	8306	3000	220	187	10:12	989	104.8
17	8000	7875	3000	220	188	10:06	1002	102.5
18	7000	6466	3000	180	2160*	6:30	1035	95.5
	149,538	141,287	54,000	2,617	4,436	138:51		

*Quantity drained from unit
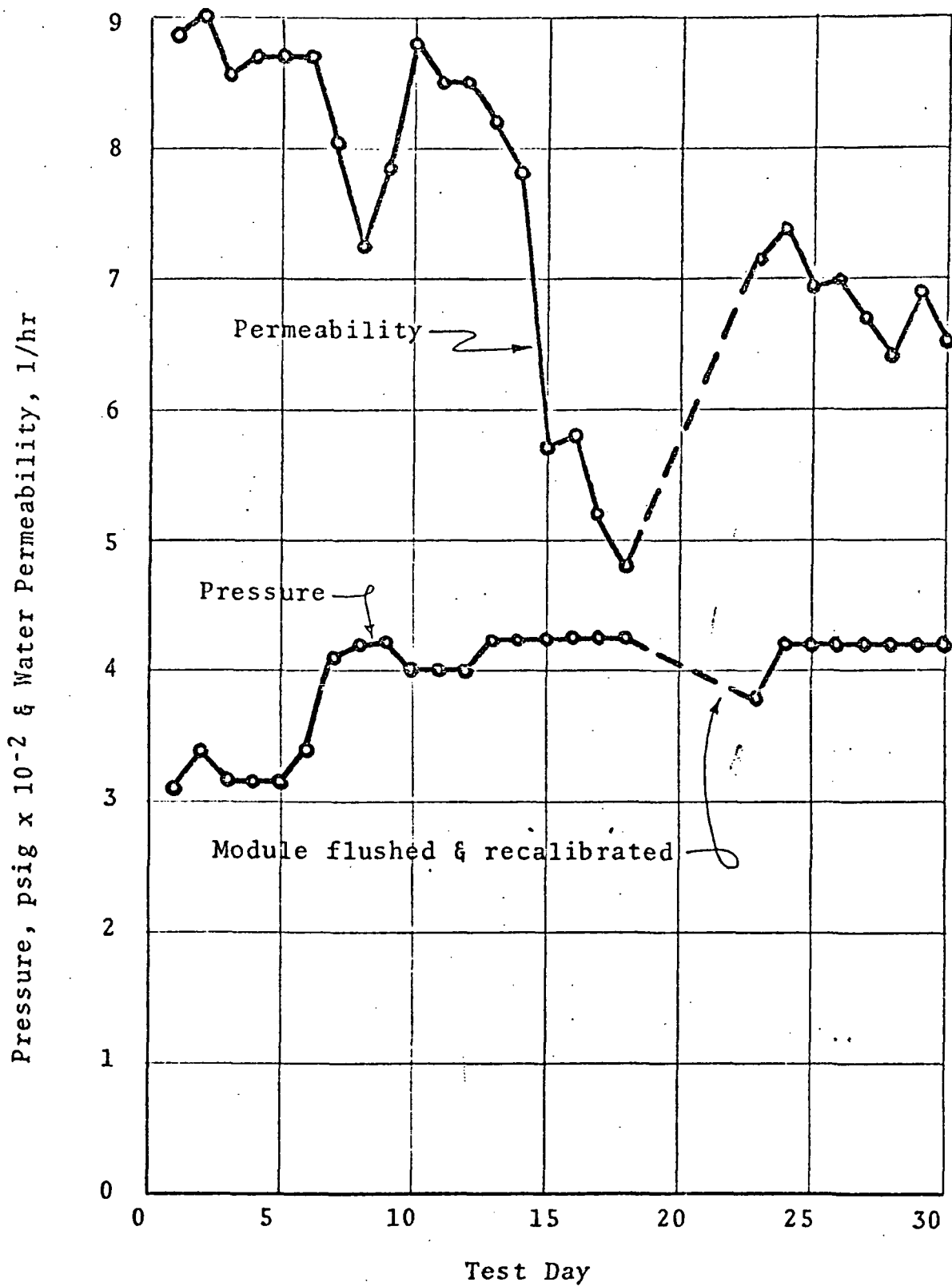
Figure 30 HYDRAULIC OPERATION OF WESTINGHOUSE MODULE

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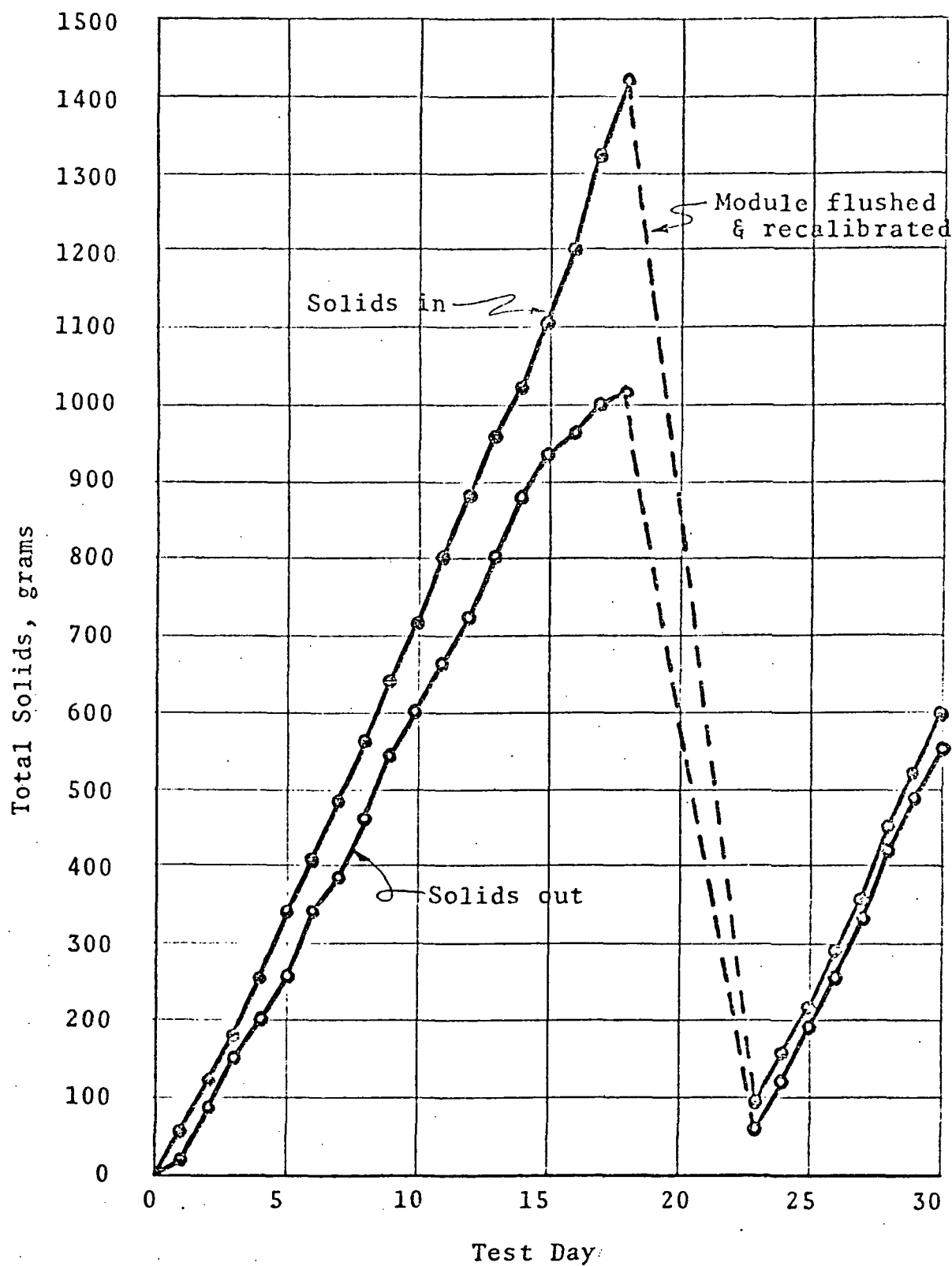
Figure 31 CUMULATIVE SOLIDS DURING FINAL WASH-LOOP TEST

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3. Flush-out of the RO unit during days #20 and 21 with tap water at a rate of approximately 30 liters/hr did remove some of the accumulated solids and restored some of the unit's capability. The permeability of the unit was increased by a factor of (7100/4770) or 1.5 as a result of this action.

4. Figure 31 illustrates the difference in performance of the RO Processor between the two series of tests. During the flush/wash water loop test (days #1 to 18 inclusive) when the yield averaged 91.2%, the difference between the solids in and the solids out became greater every day. However, during the RO waste tests (days #23 to 30 inclusive) when the yield averaged 76.7%, the difference between the solids in and the solids out remained relatively constant; the operating lines are practically parallel. The increased quantity of brine flow and/or the higher velocity maintained balanced conditions; the increased quantity of available water reduces precipitation and increases flushing.

5. The Distillation Unit's mass yield was (100 x 141,287/149,538) or 94.3%; the water yield, however, was higher (100 x 141,287/149,538 - 2 x 18 x 13.5 - 925.6) or 95.4%. The average recovery rate was 141,287/138.85 or 1018 gm/hr (2.24 lb/hr), but ranged from 1640 gm/hr (high) to 970 gm/hr (low). The recovery rate, as expected, decreased as the solids content in the Distillation Unit's recycle loop increased. Also, as expected, the specific energy increased as the solids content in the Distillation Unit's recycle loop increased; the specific energy ranged from 54.8 watt-hr/kg (low) to 112.2 watt-hr/kg (high).

6. The average purge loss was 100 x 2617/149,538 or 1.75% (based on total mass input). Daily purge losses varied considerably due to "over trimming" the purge valve.

10.5.2 Expendables Required

This test was too short to demonstrate that the useful lives of the Recycle Filter (RF), Biological Filters (BF), and the Activated Carbon Filter (ACF) are greater than 30 days as had been evidenced during the High Solids Test. However, this test demonstrated the following:

1. The useful life of the AgCl column, which had been in use since the beginning of this program, is greater than 520 + 18 x 2 or 556 man-days.

2. The useful life of the 3-micron RO prefilter is 26 x 2/8 or 6.5 man-days.

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10.5.3 Water Quality

The analytical work performed during this test was concentrated upon determining (1) the total solids and (2) organic content of the RO process stream and the distillation unit's condensate. Total solids of the distillation unit's recycle liquor was also determined.

10.5.3.1 RO Processor

Table 27 lists the daily water quality characteristics that were determined while processing used (soiled) wash water and Figure 32 depicts the day-to-day variations in permeate quality. The tabulated data and the graph show that during the flush/wash water loop test (days #1 to 18 inclusive) the permeate quality declined progressively with time, just as the hydraulic permeability. The permeate's average total solids was 10^4 x 96.4/927.4 or 104 ppm but ranged from 40 to 200 ppm. The organic content ranged from 38 to 580 ppm COD and the specific resistivity ranged from 16,900 (high) to 6,500 ohm-cm (low). The overall solids passage was 100 x 96.34/1421.9 or 6.77% - or the solids rejection was 93.2%.

From the tabulated information and Figure 32, it is apparent that during the RO waste tests (days #23 to 30 inclusive) the permeate quality improved and was better than in the loop-test. The permeate's average total solids was 10^4 x 23.3/345.7 or 68 ppm, but ranged from 40 to 120 ppm. The organic content ranged from 215 to 250 ppm COD and the specific resistivity was always above 11,000 ohm-cm. The overall solids passage was 100 x 23.3/596.1 or 3.94% - or the solids rejection was 96.1%.

As shown by the turbidity measurements, the 3-micron filtered feed contained suspended solids and colloids. Particulate accumulation fouled the membrane surface, and affected the RO processor's performance and permeate quality. There was no evidence of membrane degradation by substances in the feed; the solids rejection was 93.2 and 96.1% respectively in the two series of tests. The solids rejection of the unit ranged between 91.5 and 93.5% during the prefatory (compatibility) tests.

Permeate that was AgCl dosed always was sterile. Biological contamination was observed in the other process streams on all days tested. The MPN determination indicates the following levels of contamination.

Process Stream	Microorganisms/ml
Feed	10^7 - 10^8
Brine	10^7 - 10^8
Raw Permeate	10^3 - 10^4

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Table 27   RO PROCESSOR WATER QUALITY CHARACTERISTICS

Day & Stream	pH	Turbidity (JTU's)	Specific Resistivity (kΩ-cm)	COD (ppm)	Total Solids %
Day No. 1					
Feed	8.25	65	2.6	2020	0.119
Filtered Feed*	8.2	50	2.7	1500	0.108
Brine	8.75	48	0.3	12240	0.884
Permeate	9.5	0.28	12.3	38	0.004
Day No. 2					
Feed	8.6	54	2.4	1950	0.125
Filtered Feed*	8.6	38	2.4	1900	0.112
Brine	9.0	42	0.1	42000	2.89
Permeate	9.9	0.10	9.9	104	0.004
Day No. 3					
Feed	8.7	52	2.4	2150	0.127
Filtered Feed*	8.7	22	2.4	1800	0.111
Brine	9.15	25	0.2	26000	1.489
Permeate	9.9	0.05	10.0	160	0.006
Day No. 4					
Feed	8.85	150	2.0	2600	0.155
Filtered Feed*	8.8	80	2.0	2150	0.125
Brine	9.15	100	0.2	20000	1.430
Permeate	10.2	0.15	8.6	120	0.010
Day No. 5					
Feed	8.4	150	2.1	3800	0.167
Filtered Feed*	8.4	52	2.1	2080	0.129
Brine	9.1	55	0.2	27000	1.804
Permeate	10.05	0.12	9.2	190	0.007
Day No. 6					
Feed	8.4	90	2.0	2400	0.166
Filtered Feed*	8.4	38	2.0	2050	0.131
Brine	9.0	15	0.2	37000	1.902
Permeate	9.8	0.5	9.6	220	0.012
Day No. 7					
Feed	7.9	130	1.9	2600	0.189
Filtered Feed*	7.8	75	1.9	2000	0.127
Brine	8.75	75	0.3	14000	0.998
Permeate	9.6	1.0	10.6	260	0.005

* 3 micron

C H E M T R I C

Table 27 (Continued)

Day & Stream	pH	Turbidity (JTU's)	Specific Resistivity (kΩ-cm)	COD (ppm)	Total Solids %
Day No. 8					
Feed	8.1	120	1.8	3000	0.176
Filtered Feed*	8.1	100	1.8	2500	0.149
Brine	8.6	130	0.4	8000	0.707
Permeate	8.9	0.48	16.9	301	0.006
Day No. 9					
Feed	8.2	95	2.3	2700	0.164
Filtered Feed*	8.2	70	2.3	2150	0.126
Brine	8.7	45	0.4	18000	0.917
Permeate	9.3	1.2	11.8	340	0.005
Day No. 10					
Feed	8.4	100	2.5	3000	0.180
Filtered Feed*	8.4	40	2.5	2150	0.132
Brine	8.7	30	0.2	25600	1.566
Permeate	9.6	0.30	12.1	310	0.014
Day No. 11					
Feed	8.5	140	2.2	2800	0.202
Filtered Feed*	8.5	30	2.2	2250	0.142
Brine	8.8	35	0.2	20800	1.285
Permeate	9.7	0.65	11.6	310	0.010
Day No. 12					
Feed	8.55	150	2.2	2900	0.193
Filtered Feed*	8.55	28	2.2	1800	0.144
Brine	8.8	25	0.2	21600	1.463
Permeate	9.8	0.30	10.2	280	0.010
Day No. 13					
Feed	8.15	125	2.1	5200	0.210
Filtered Feed*	8.15	50	2.1	2100	0.136
Brine	8.5	30	0.3	17600	1.080
Permeate	9.3	2.0	9.0	410	0.016
Day No. 14					
Feed	8.1	120	2.1	5900	0.211
Filtered Feed*	8.1	40	2.1	2050	0.112
Brine	8.1	40	0.3	16000	0.915
Permeate	8.7	1.0	10.1	410	0.019

* 3 micron

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Table 27 (Continued)

Day & Stream	pH	Turbidity (JTU's)	Specific Resistivity (kΩ-cm)	COD (ppm)	Total Solids %
Day No. 15					
Feed	8.2	150	2.0	3600	0.213
Filtered Feed*	8.2	130	2.0	2850	0.150
Brine	8.2	210	0.5	12800	0.640
Permeate	8.3	4.5	10.0	470	0.020
Day No. 16	8.2	120	1.6	3300	0.226
Filtered Feed*	8.1	65	1.6	3200	0.190
Brine	8.25	75	0.6	8400	0.459
Permeate	9.0	0.30	10.8	450	0.015
Day No. 17					
Feed	8.45	100	1.6	3400	0.225
Filtered Feed*	8.3	42	1.2	3300	0.212
Brine	8.4	50	0.5	10400	0.529
Permeate	9.2	2.5	6.5	580	0.015
Day No. 18					
Feed	8.45	85	1.5	2900	0.209
Filtered Feed*	8.25	48	1.4	2650	0.188
Brine	8.4	77	0.6	16000	0.414
Permeate	9.2	2.0	7.4	490	0.009
Day No. 19 Calibration					
Day No. 20 Tap Water Flush-out					
Day No. 21 Tap Water Flush-out					
Day No. 22 Recalibration					
Day No. 23					
Feed	8.3	80	1.9	3800	0.215
Filtered Feed*	8.1	46	1.9	2450	0.157
Brine	8.2	45	0.6	9200	0.535
Permeate	8.65	0.10	11.1	215	0.012

* 3 micron

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Table 27 (Continued)

Day & Stream	pH	Turbidity (JTU's)	Specific Resistivity (kΩ-cm)	COD (ppm)	Total Solids %
Day No. 24					
Feed	8.5	85	2.8	4800	0.156
Filtered Feed*	8.25	45	2.7	1800	0.117
Brine	8.3	43	0.6	10800	0.546
Permeate	8.7	0.25	14.4	225	0.003
Day No. 25					
Feed	8.5	85	2.5	3000	0.183
Filtered Feed*	8.2	28	2.5	2800	0.109
Brine	8.4	80	0.6	10800	0.582
Permeate	9.0	0.06	11.8	230	0.003
Day No. 26					
Feed	8.5	85	2.5	3100	0.153
Filtered Feed*	8.3	30	2.5	2800	0.123
Brine	8.4	70	0.7	10900	0.523
Permeate	9.4	0.40	11.6	223	0.011
Day No. 27					
Feed	8.3	120	2.3	2800	0.156
Filtered Feed*	8.2	20	2.3	2000	0.138
Brine	8.25	60	0.7	9600	0.540
Permeate	9.35	0.30	13.0	245	0.005
Day No. 28					
Feed	8.7	85	2.1	5100	0.215
Filtered Feed*	8.7	25	2.2	2300	0.155
Brine	8.8	55	0.8	8400	0.489
Permeate	9.6	0.32	16.5	250	0.009
Day No. 29					
Feed	8.10	85	2.5	3800	0.176
Filtered Feed*	8.05	28	2.5	1950	0.122
Brine	8.50	58	0.8	6400	0.435
Permeate	8.45	1.0	16.3	230	0.006
Day No. 30					
Feed	8.1	180	2.5	3850	0.253
Filtered Feed*	8.1	35	2.6	1950	0.136
Brine	8.3	95	0.9	9200	0.407
Permeate	8.35	0.60	17.5	245	0.004

* 3 micron
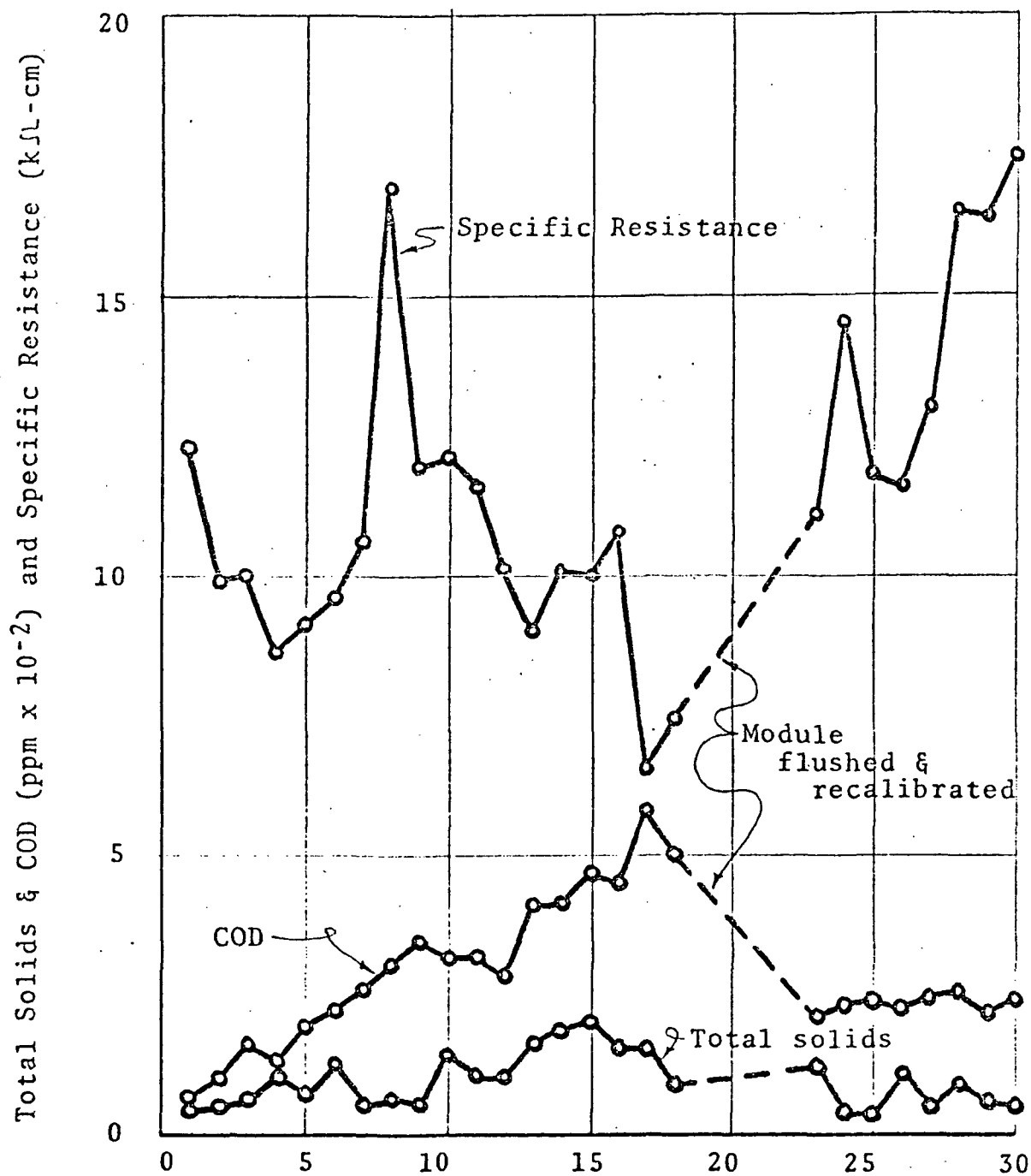
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Figure 32   PERMEATE QUALITY CHARACTERISTICS

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No adverse effects were experienced by the test crew using Neutrogena Rainbath Body Soap Gel and renovated wash water for showering. In general the renovated water was not "sudsy" and rinsing was better than previously experienced during the other wash-loop tests.

10.5.3.2 Distillation Unit

Table 28 lists the daily water quality characteristics of raw condensate and recycle liquor during the loop test. As expected the quality of the condensate declined as the solids content of the distillation unit's recycle loop increased.

The distillate (raw condensate) produced when processing RO brine and commode fecal flush is approximately 3 times greater in organic content than the distillate produced when processing urine and urinal flush water. RO brine contains about ten parts of organic solids, whereas, urine contains about 2 parts of organic solids to one part of inorganic solids.

10.5.4 Recalibration and Inspection

Since the distillation unit's electric motor failed, the unit could not be recalibrated. On the 19th test day the distillation unit was disassembled and inspected. The evaporator was found to be clean as usual, however, the inlet to the compressor and the internal surfaces of the compressor were covered with a brown residue. This residue was water soluble and obviously was "dried-out" recycle liquor that was entrained with the water vapor and had subsequently "dried-on" the internal surfaces.

After soaking-off the residue, close inspection of the compressor showed that some of the buff sealer was loose on one of the rotors. Tests on the bench fixture, however, showed that the compressor drew the same vacuum as it did originally before the start of the loop test.

The RO Processor was recalibrated on days 19 after termination of the flush/wash water loop test, on day 22 again after tap water flush-out, on day 31 again after completion of the RO waste test, and again on days 33 and 36 after additional tap water flush-outs. Figure 33 presents the results of these calibrations and/or recalibrations. Deionized tap water was used in the calibration and/or recalibration to aleviate the effects of concentration polorization.

Figure 33 shows that the RO unit had lost a great deal of its permeability by day 19, during the flush/wash water loop tests. Tap water flush-outs at a rate of 30 liters/hour on days 20 and 21 restored a significant portion of units capability as indicated by the 22nd day recalibration curve.

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Table 28   DISTILLATION UNIT WATER QUALITY CHARACTERISTICS

Day & Stream	pH	Turbidity (JTU's)	Specific Resistivity (kΩ-cm)	COD (ppm)	Total Solids %
Day No. 1					
Condensate	4.5	1.8	20.5	28	0.001
Day No. 2					
Condensate	4.2	1.4	18.2	54	0.001
Day No. 3					
Condensate	7.2	3.5	30.4	80	0.0009
Recycle Liquor	---	---	---	---	7.89
Day No. 4					
Condensate	7.8	3.5	20.35	70	0.0007
Recycle Liquor	---	---	---	---	8.980
Day No. 5					
Condensate	7.7	8.5	14.2	90	0.001
Recycle Liquor	---	---	---	---	12.65
Day No. 6					
Condensate	7.9	20.0	15.4	115	0.003
Recycle Liquor	---	---	---	---	15.287
Day No. 7					
Condensaate	8.5	10.0	11.8	150	0.0005
Recycle Liquor	---	---	---	---	16.07
Day No. 8					
Condensate	8.6	5.4	9.8	250	0.0003
Recycle Liquor	---	---	---	---	19.036
Day No. 9					
Condensate	7.95	4.0	11.4	230	0.0004
	---	---	---	---	19.043

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Table 28 (Continued)

Day & Stream	pH	Turbidity (JTU's)	Specific Resistivity (kΩ-cm)	COD (ppm)	Total Solids %
Day No. 10					
Condensate	8.6	5.5	9.2	240	0.0089
Recycle Liquor	---	---	---	---	22.40
Day No. 11					
Condensate	8.8	2.9	10.0	170	0.002
Recycle Liquor	---	---	---	---	20.30
Day No. 12					
Condensate	8.7	8.5	11.2	80	0.002
Recycle Liquor	---	---	---	---	20.26
Day No. 13					
Condensate	8.3	6.4	9.4	280	0.001
Recycle Liquor	---	---	---	---	19.32
Day No. 14					
Condensate	7.8	5.4	10.5	310	0.002
Recycle Liquor	---	---	---	---	19.83
Day No. 15					
Condensate	8.5	5.5	6.8	380	0.002
Recycle Liquor	---	---	---	---	22.40
Day No. 16					
Condensate	7.2	2.0	14.3	380	0.0004
Recycle Liquor	---	---	---	---	22.36
Day No. 17					
Condensate	7.7	5.2	7.0	390	0.001
Recycle Liquor	---	---	---	---	22.70
Day No. 18					
Condensate	8.2	8.0	7.8	140	0.001
Recycle Liquor	---	---	---	---	---

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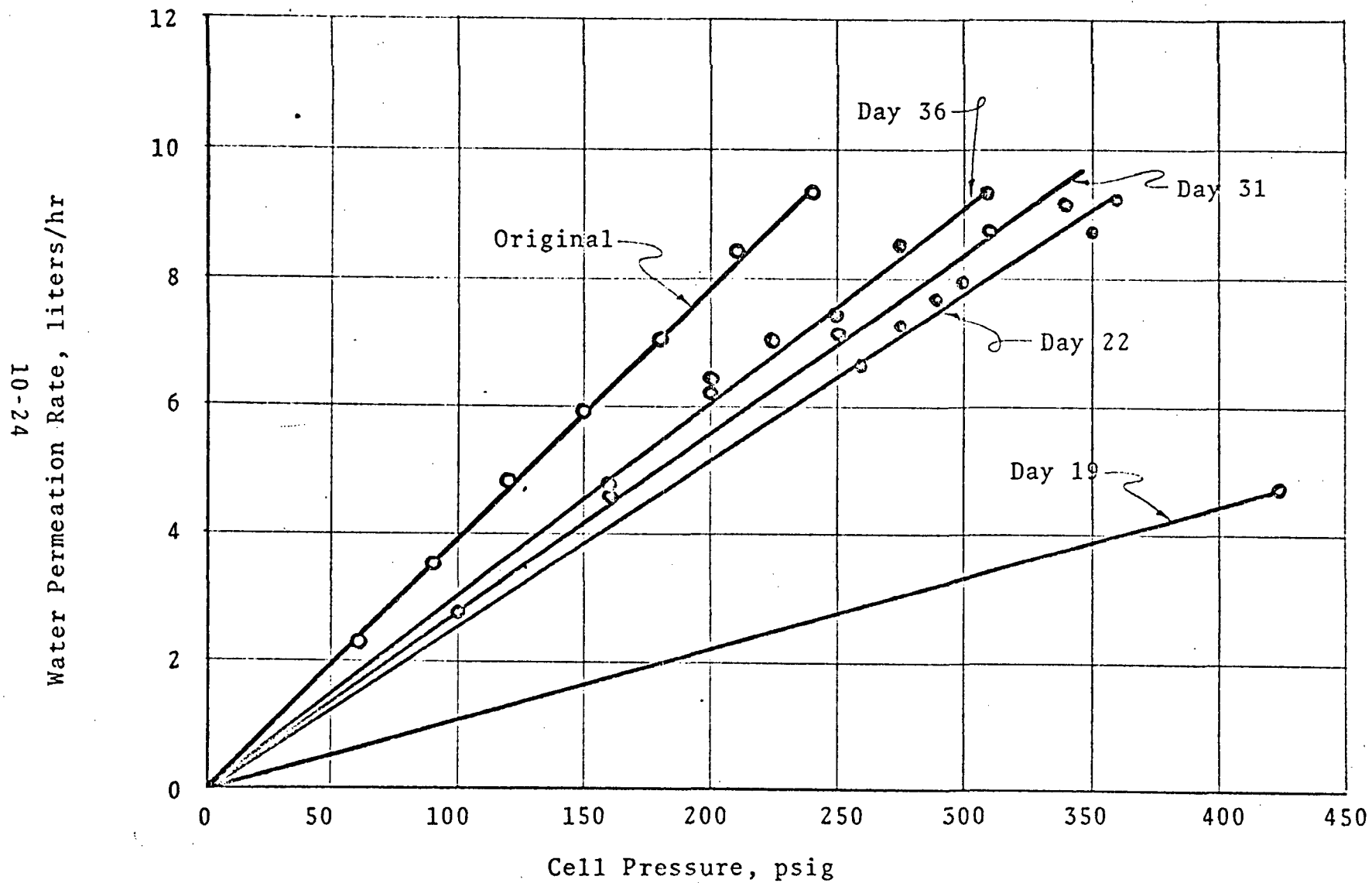
Figure 33   CALIBRATION CURVES FOR THE WESTINGHOUSE MODULE

During the RO waste tests (days #23 to 30 inclusive) there was no significant build-up of "fouling" solids in the unit as evidenced by the materials balances presented in Table 25 and in Figure 29. The data in Figure 31, namely, the 31st day curve, shows no loss in permeability. Furthermore, it appears that the unit was cleansing itself further with the increased brine flow, during this test series when yield averaged 76.7%.

Further tap-water flush-outs were performed on days 32, 34 and 35 at a flow rate of approximately 120 liters/hr. The 36th day curve on Figure 31 shows that more of the unit's permeability was restored by the higher velocity flush-outs.

No attempt was made to quantify the solids collected during the flush-outs because they were usually unattended. The solids collected, however, can be characterized as flaky, greasy, gray precipitates with a specific gravity greater than one.

10.6 Conclusions and Recommendations

From the results obtained during this test the following were concluded.

1. Westinghouse's 4 x 4 Basic Module performs satisfactorily when operated at water yields of 75% or less. Solids accumulation (fouling) will be minimal or insignificant and therefore not affect the units hydraulic characteristics (operating pressure and permeability). The raw permeate will only require AgCl dosing for sterility and activated carbon filtration to reduce the COD from approximately 250 ppm to 100 ppm.

2. Westinghouse's 4 x 4 Basic Module when operated at water yields of 90 - 95% will not perform satisfactorily. Solids accumulation (fouling) will occur, and the hydraulic characteristics and permeate quality will be adversely affected.

3. Westinghouse's membrane was not degraded by substances in the feed after 60 days of exposure at pH's ranging between 8.1 and 10.2.

4. The quantity of germicide employed (ca 400 ppm Vancide BN) was not adequate to maintain sterility. All of the RO process streams were biologically contaminated.

5. The useful life of the 3-micron filter upstream of the RO feed pump is 6 man-days.

This test demonstrated that Westinghouse's 4 x 4 Basic Module does not have the useful life desired when operated under current SSP concepts and criteria (eg., 95% yield). The basic problem is surface fouling. It is recommended that increases in flow by recycle and/or periodic flush-outs be investigated to control the unit's permeability and subsequently its service life.

This test also demonstrated that the volumetric efficiency of a "tight dry-coated" compressor can be equal to that of a lubricant coated compressor. However, this test failed to demonstrate that the applied coating had the useful life desired; the coating became loose. It is recommended that surface preparation be investigated to increase coating durability.

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PRELIMINARY REVERSE OSMOSIS INVESTIGATIONS

Previous work on reverse osmosis (RO) has indicated that cleansing agents have a significant effect on membrane performance and useful life. Since currently available RO theories cannot predict the effect of the organic solutes found in cleansing agents - it was necessary to experimentally evaluate the effect of candidate surfactants and germicides on currently available RO membranes.

A.1 Previous Work

The Aerojet-General Corporation under subcontract to The Marquardt Corporation performed research and development on a reverse osmosis wash-water recovery system, during January to June 1965. The criteria established by Marquardt are as follows.

A. Process 10.5 gallons/day of a wash-water solution containing 2000 ppm of dissolved solids, including detergents and waste materials.

B. Recover 95% of the water as product.

C. The recovered water shall (1) meet USPHS Standards for Drinking Water, (2) contain no more than 1.0 ppm of ammonia, (3) have a pH ranging from 5.0 to 8.5, and (4) contain less than 2.2 microorganisms per millimeter.

D. The prototype system developed shall be representative of a flight-qualifiable model.

During experimental investigations with one of their own membranes, Aerojet-General observed flux-depressant effects when processing a feed solution containing 10,000 ppm of Steraskin (an antiseptic skin cleanser formulation from Chas. Pfizer and Co., Inc.). Permanent losses in membrane flux were experienced - ranging from 40 to 80% (increasing with operating pressure) within four hours. Two ingredients in the Steraskin formulation - namely, Hexachlorophene and 4-chloro - 3, 5, - Xylenol - were found to be the flux depressants. From this work Aerojet-General concluded that an RO system requires a detergent formulation that does not have any flux depressant. Furthermore, in the absence of a definite detergent selection, Aerojet-General recommended suspension of further work on flight-hardware because design criteria could not be determined until a cleansing agent was selected.

A.2 Cleansing Agents

The following discussion is presented to indicate the rationale used in selecting candidate cleansing agents.

Cleansing agents such as soaps and detergents for toilet, household and industrial applications are formulations composed of surface-active agents and builders. Surface-active agents (surfactants) include soaps, detergents, emulsifiers, wetting agents and penetrants. They modify the properties of the surface layer between two phases in contact with each other. Most surfactants are a combination of water-attracting (hydrophilic) groups, with water-repelling (hydrophobic) groups. Surfactants increase wetability; the hydrophobic group attaches itself to the grease, fat, or oil to be solubilized, leaving the hydrophilic group free to attract water.

Soap comprises the sodium and potassium salts of various natural fats, oils and/or acids - but chiefly oleic, stearic, and palmitic acids. Today, data shows that soap accounts for only 16.8% of the market and synthetic detergents (syndets) have 83.2% of the market. The syndets surpass soap in wetting-out, emulsifying, and detergent properties - and they do not hydrolyze to alkali in water. Syndets are stable towards acids, alkalies and salts - but most important of all they are not precipitated by the hardness (Ca and Mg ions) present to some extent in most water.

Surfactants are classified by type - namely, (1) anionic, (2) nonionic, (3) cationic, and (4) amphoteric. The anionic surfactants are essentially detergents and comprise the sulfonates, sulfates and sodium salts of alkylbenzenes, alaphatic alcohols, and alaphatic ethers. The nonionic surfactants are used as detergents, wetting agents, emulsifiers, and/or dispersants, and essentially comprise the products of ethylene and its homologues and the complex block polymers. Cationic surfactants are used as softeners, germicides, antisats, and/or emulsifiers - and comprise the products of tertiary amines, alkyl amines, alkylbenzyl amines or amonium chlorides. The amphoteric surfactants are used as penetrants, wetting agents, foaming agents and/or detergents - and comprise amines and salts of dicarboxylic fatty acid derivatives, ethoxylated amine condensates, alkyl ammonium sulfonic acid betaines, aryl betaines and/or alkyl amido betaines.

While soap and syndets are the primary cleansing agents, their use alone does not provide the best results. Except for the purest of toilet bars, various chemicals are added as "builders" to improve the overall cleansing quality. Practically all bars contain from 10 to 30% water; if the bar was anhydrous, it would be too hard to dissolve easily. Almost all toilet bars contain perfume to disguise the original odor. Polymerized toluene sulfonate salts are used to control sloughing of the bar, and to increase free rinsing. High molecular weight ethylene polymers are used as slip agents. Sodium bicarbonate is often used as a mild alkali builder. Bleached white milled wheat flour,

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cornstarch, or titanium dioxide are used as abrasive agents. Germicidal agents are used to provide disinfecting properties.

In laundering or heavy-duty cleaning, various pH "builders" are added to improve the washing results because heavy soils, in general, represent a strong neutral buffer material that is acidic to soap or syndets. Caustic soda, soda ash, sodium tripolyphosphate, tetra potassium pyrophosphate, sodium metasilicate, sodium orthosilicate, sodium sesquisilicate, and/or modified soda are typical alkali builders. Perborates or cyanuric acid derivatives are used as bleaching agents. Quaternary salts of aromatic cyclic imides are used as germicides. Minor ingredients include bluing agents, optical whitening agents, fluorescent dyes, perfume and soil suspending agents.

The list of syndets, wetting agents, emulsifiers, penetrants, and dispersants is quite formidable - perhaps over 1000.*

It is obvious from this discussion that a particular formulation for a particular application (e.g., personal hygiene and laundering) would consist of several ingredients and may be compounded from a variety of available materials. It can be predicted that electrolytes and suspended solids (i.e., the inorganic alkali builders, and the whitening and/or abrasive agents) would be readily rejected by reverse osmosis membranes and upstream filters - and therefore, do not have to be considered for experimental membrane-compatibility determinations. The remaining constituents are classified by type; namely, (1) anionic, (2) nonionic, (3) cationic, and (4) amphoteric.

The following examples of each class were selected for membrane-compatibility investigations. These materials are used in formulating skin cleansers, hair shampoos, pharmaceuticals, dentifrices, medicated cosmetics, germicidal cleansers, and/or light-duty detergents for dishwashing and washing of fine fabrics.

Anionic

Mild Soap   - Castile (Fisher Scientific Co.)

Detergent   - STEPHANOL WA-100, sodium lauryl sulfate (Stepan Chemical Co.)

Detergent   - BIOTERGE AS-35CL, alpha olefin sulfonate (Stepan Chemical Co.)

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*McCutcheson, J. W., "Detergents and Emulsifiers", J. W. McCutcheson, Inc., Morristown, N. J., 1967 Annual

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Detergent & Conditioner	- MAYPON 4 CT, triethanolamine salt of a cocyl- polypeptide condensate (Stepan Chemical Co.)
Detergent	- STEOL 4N, ethoxylated fatty alcohol sulfate (Stepan Chemical Co.)
Detergent & Conditioner	- MAYPON 4C, potasium salt of the condensation product of coconut fatty acids with a com- plex of polypeptides and amine acids (Stepan Chemical Co.)
Antibacterial Skin Cleanser	- pHisoHEX, sodium octylphenoxyethoxyethyl ether sulfonate and Hexachlorophene (Win- throp Laboratories, Div. of Sterling Drugs)
Germicide	- VANCIDE BN, sodium bithionolate (R. T. Van- derbilt Co.)

Nonionic

Detergent	- BIO SOFT EA-10, linear alkoxyalkylene eth- anol (Stepan Chemical Co.)
Germicide	- PVP-Iodine 10, polyvinylpyrrolidone-iodine complex (General Aniline & Film Corp.)

Cationic

Germicide	- HYAMINE 3500, blend of alkyl dimethyl benzyl ammonium chlorides (Rohm and Haas Co.)
Germicide	- HYAMINE 10-X, diisobutyleresoxyethoxyethyl dimethyl benzyl ammonium chloride (Rohm and Haas Co.)
Germicide	- BTC 2125, 1:1 mixture of alkyl dimethyl benzyl ammonium chlorides and alkyl dim- ethyl ethylbenzyl ammonium chlorides (Onyx Chemical Co.)
Germicide	- Hyamine 1622, diisobutylphenoxyethoxyethyl dimethyl benzyl ammonium chloride (Rohm and Haas Co.)

Amphotheric

Detergent	- MIRANOL C2M, dicarboxylic coconut derived diethanolamine salt (Miranol Chemical Co.)

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A.3 Membranes

Practical reverse osmosis membrane materials must exhibit the following characteristics:

A. Salt rejection capability in excess of 90%.

B. Water permeabilities in the range 5 to 25 $gal/ft^2/day$.

The literature indicates that only a few materials have salt rejection capability greater than 90%; namely, (1) cellulose acetate, (2) ion exchange membrane materials, (3) cross-linked polyvinyl alcohol, (4) ethyl cellulose, (5) polyurethane, and (6) nylon. To exhibit the desired hydraulic permeability, the above polymeric materials must be modified and/or fabricated into ultrathin films.

When this investigation was started, five different materials had been developed which showed promise for wash water reclamation. These materials are described as follows.

A. Cellulose Acetate - The bulk of the present reverse osmosis technology is predicated on the properties of cellulose acetate as modified by the procedures of Loeb and Sourirajan. Several grades are available, ranging from high selectivity and low flux to low selectivity and high flux. A partial list of manufacturers who supply cellulose acetate membranes or units with cellulose acetate membranes is given below.

    Aerojet-General
    American Standard Corporation
    Culligan
    Gulf-General Atomics
    Eastman Chemical Products
    Havens Industries
    Universal Water Corporation

B. Blend CA - Aerojet-General has prepared membranes from a blend of cellulose diacetate and triacetate which are superior to the cellulose acetate membranes in regard to both salt rejection and compaction resistance.

C. CAB - Universal Water Corporation has prepared membranes from cellulose acetate butyrate which exhibit better compaction resistance but lower hydraulic permeability than the cellulose acetate membranes.

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D.   Permasep B-5* - E. I. DuPont deNemours & Company has devel-
     oped a "Permasep" permeator which utilizes hollow fiber ma-
     terials; i.e., the membranes are heavy walled cylinders of
     very small diameters (approximately 50 to 150 microns).
     Although DuPont does not disclose the chemical composition
     of its "Permasep" materials, it is surmized that the base
     polymer is nylon.

E.   Diaflo** - Amicon Corporation has prepared ultrafiltration
     membranes from polyelectrolyte complexes ("polysalts").
     These membranes are employed in concentration, purification
     and separation of macromolecules. They exhibit high water
     flux at modest pressures (10 - 100 psi). Although Diaflo
     membranes are not classified as RO membranes, since they
     don't effectively retain electrolytes, it appears that these
     membranes offer possibilities for the separation of organic
     solutes down to the 200 molecular weight range.

All of these membrane materials were selected for experimental
evaluation during this program.

A.4 Experimental Investigations

     The experimental research performed was divided into two
tasks - namely, screening tests and parametric tests. The results
of the screening tests were used to select materials for the par-
ametric tests.

     A.4.1 Test Apparatus

          A test cell was designed and fabricated according to
the Office of Saline Water guidelines,*** and assembled with an-
cillary components into a feed and bleed processing system. The
test cell accommodates a 2-1/2" x 5-1/2" sample of film (effect-
ive area is 1-1/2" x 3-1/2") and provides a rectangular flow chan-
nel (2" x 0.006"). The test cell is constructed from aluminum,
and coated with a fluorocarbon resin (Kynar) to resist corrosion.
A photograph of the test cell is presented on the next page.

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*   DuPont's registered trademark for it's permeators.
**   Amicon's registered trademark for it's membranes.
*** Podall, H.E., et al., "Recommended Procedures for Testing and
     Evaluation of Candidate Reverse Osmosis Membranes for Desal-
     ination", OSW R&D Progress Report No. 268, Sept. 1967.

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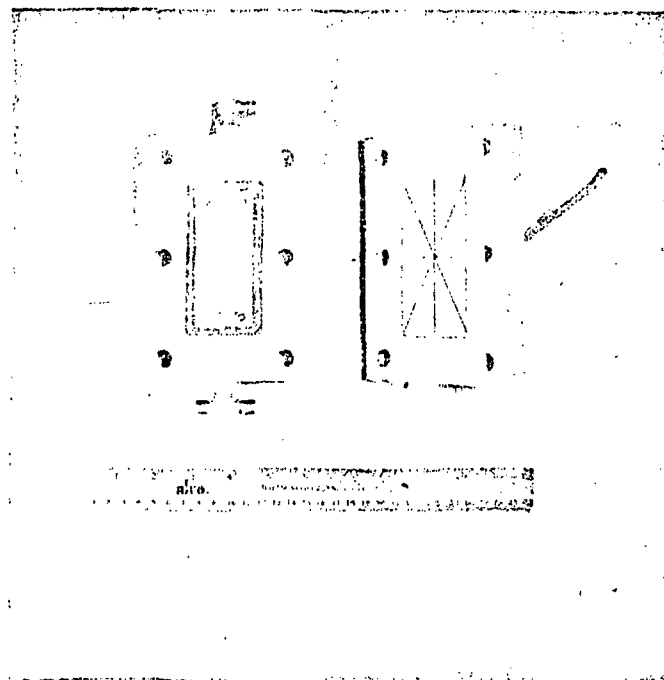
MEMBRANE (NOT SHOWN)
IS LOCATED BETWEEN
RECTANGULAR PLATES
AND IS SUPPORTED
BY A SINTERED PANEL.
PERMEATE FLOWS
THROUGH THE PANEL
TO THE RIGHT-HAND
PLATE, AND EXITS
VIA CENTRAL HOLE.

Figure A-1   EXPLODED VIEW OF REVERSE OSMOSIS TEST CELL

The schematic presented in Figure A-2 illustrates the design of the test set-up. Two pumps were included in the circuit. A hydraulically activated pump (Lapp Pulsafeeder - Model LS-10) was used to feed the test apparatus and develop the operating pressure. A gear pump (Eastern Model No. 107, modified by CHEMTRIC to contain a high pressure shaft seal) was used to develop varying hydrodynamic conditions (i.e., fluid velocities or Reynold number) by recirculating a fraction of the brine. The gear pump was driven by a variable speed motor. Since the performance of an RO membrane is a strong function of temperature, a concentric-tube heat exchanger was incorporated into the recycle loop to remove heat generated by the gear pump, and to maintain relatively constant temperature operations.

The operating pressure was governed by the pressure on the gas dome of the back-pressure regulator. In operation, the regulator's diaphragm and nozzle formed a variable orifice to relieve the pressure pulses generated by the feed pump. The hydraulic accumulator alleviated further pressure fluctuations. A pressure relief valve was included to protect the system should a malfunction occur.

A.4.2   Screening Tests

In this task the five (5) candidate membrane materials (cellulose acetate, Blend CA, CAB, Permasep and Diaflo) were screened with candidate cleansing agent ingredients under reverse osmosis conditions. Typical examples of each class of surface active agents were selected for membrane-compatibility investigations.

A.4.2.1   Test Procedure

Processing at 600 psi in a single pass without recycle was selected as the operating condition for screening tests. Two Permasep units were tested as modules, since the fiber material used in this unit is not available in "sheet" form. In the case of sheet membrane material, a new membrane was placed in the test cell each run. The cell was initially fed test solution for two hours to establish an operating equilibrium. Brine and permeate streams were then collected to determine stream flow rates and their quality (pH, conductivity, and total solids). Usually a run was terminated when 300 - 500 ml of permeate was collected (the amount necessary to perform the analyses). In the case of Permasep permeators, the units were flushed-out for four (4) hours with deionized tap water in between tests.

The setting of the hydraulically activated diaphragm feed pump was adjusted to maximize the feed rate (ca. 0.6 gph). Test solutions were composed of a single solute dissolved in deionized

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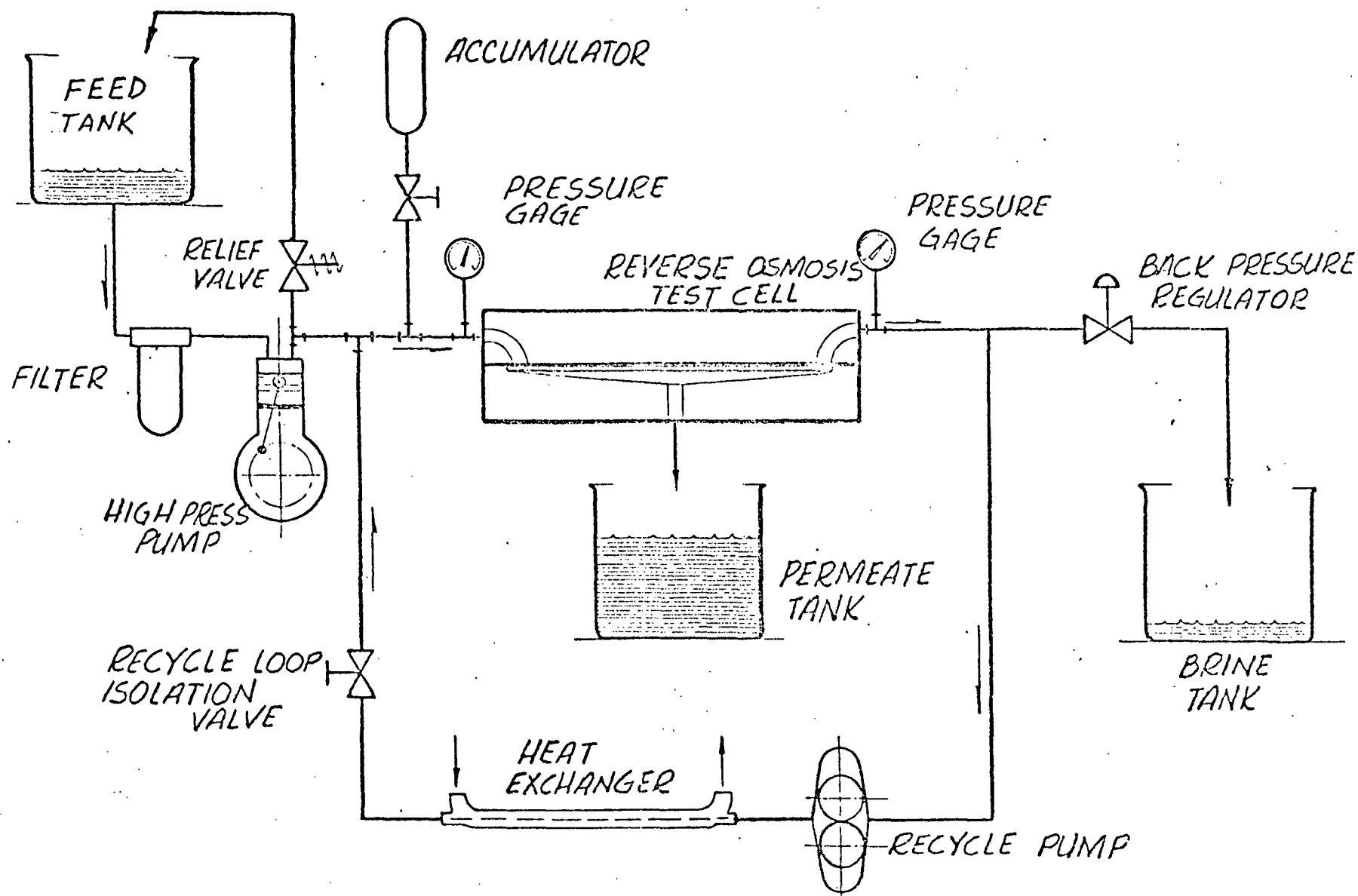
Figure A-2   FLOW SCHEMATIC FOR REVERSE OSMOSIS SCREENING TESTS

tap water, rather than a mixture - to ascertain compatibility of the ingredient, avoid synergestic effects and minimize subsequent analytical work.

A.4.2.2 Discussion of Results

The results presented in Table A-1 thru A-7 indicate that most of the membranes can reject more than 90% of the dissolved organic solutes. Only one membrane material, namely Diaflo UM-05 (see Table 5) was chemically degraded by a surfactant - i.e., sodium lauryl sulfate, which is a principle ingredient in dentifrice formulations.

The cellulose acetate base or derivative membrane materials exhibited excellent rejection capability for both electrolytes and organic solutes. The difference between these membranes is primarily hydraulic permeability or "tightness". The permeation rate data indicates that Eastman's RO-97 membrane is (1) approximately 1.5 times faster than Gulf General Atomics ROGA membrane, (2) approximately 3 times faster than Aerojet-General's Blend CA membrane, and (3) approximately 10 times faster than the Universal Water Corporation's CAB membrane.

The results presented in Table A-5 indicate that Diaflo UM-05 is more permeable than Eastman's RO-97 membrane. However, because it was chemically degraded by a common surfactant it is not considered as practical membrane material for wash water processing.

Tables A-6 and A-7 show that the DuPont Permasep units effectively reject all the selected solutes except the benzalkonium chlorides. The data indicates that their retention in a homolgous series increases with increasing molecular weight and/or with increasing branching. The slight difference in performance between these two units can be attributed to differences in their salt permeation efficiency (5% vs 3%) or internal imperfections.

The results in Table A-6 show that the flux declined considerably when Maypon 4C was processed. It appears that the BTC 2125 (cationic agent) from the previous run was not flushed out adequately and this residual reacted with the Maypon 4C (anionic agent) to form a sol or precipitate in the shell side of the Permasep permeator. The precipitate deposited on the hollow fibers and fouled the unit. Bench tests subsequently confirmed the precipitation problem between these compounds.

Vancide BN and PVP-Iodine 10 were rejected better than the quartenary ammonium salt germicides by the Permasep units, as indicated by the results in Table A-7. DuPont discourages the use of iodophors because iodine reacts with the B-5 Permasep polymer material.

C H E M T R I C

Table A-1   SCREENING PERFORMANCE OF EASTMAN'S RO-97 MEMBRANE

Solute	Permeation Rate (ml/hr)	Concentration		Rejection (%)
		Feed (ppm)	Permeate (ppm)	
Deionized Water Only	183			
Sodium Chloride	168	1536	200	86.2
Castile Soap	63	1340	40	97.0
MIRANOL C2M	190	1776	46	97.5
HYAMINE 10-X	178	494	15	97.0
VANCIDE BN	135	451	⊰1	99.9+
HYAMINE 3500	165	553	50	90.9
PVP-Iodine 10	164	421	20	95.2
STEPANOL WA-100	69	1471	142	90.3
BIOTERGE AS-35CL	127	1324	16	98.8
MAYPON 4CT	149	1562	48	96.9
STEOL 4N	110	1456	45	96.9
MIRANOL C2M	144	8883	576	93.5
HYAMINE 3500	149	4736	44	99.1
Mixture - 10 parts MIRANOL C2M to 1 part NaCl	159	11801	1133	90.4
pHisoHex	103 to 54 (See Fig.A-3)	532	10	98.1

C   H   E   M   T   R   I   C

Table A-2 <u>SCREENING PERFORMANCE OF GENERAL ATOMIC'S ROGA MEMBRANE</u>

Solute	Permeation Rate (ml/hr)	Concentration		Rejection (%)
		Feed (ppm)	Permeate (ppm)	
Deionized Water Only	110			
Sodium Chloride	116	1452	59	95.7
MIRANOL C2M	117	1862	11	99.3
HYAMINE 1622	103	1499	2	99.8
Castile Soap	81	1338	15	99.0
STEPANOL WA-100	86	1728	54	96.9
HYAMINE 3500	102	1737	10	99.3
BIOTERGE AS-35CL	92	1617	8	99.3

C H E M T R I C

Table A-3 SCREENING PERFORMANCE OF AEROJET'S CA BLEND MEMBRANE

Solute	Permeation Rate (ml/hr)	Concentration		Rejection (%)
		Feed (ppm)	Permeate (ppm)	
Deionized Water Only	63			
Sodium Chloride	61	1436	67	95.3
Castile Soap	29	1340	18	98.5
MIRANOL C2M	55	1776	4	99.8
HYAMINE 10-X	50	494	39	92.1
VANCIDE BN	34	451	<1	99.9+
HYAMINE 3500	49	554	18	96.7
PVP-Iodine 10	31	421	70	83.3
STEPANOL WA-100	38	1471	53	96.4
BIOTERGE AS-35CL	36	1324	4	99.7
MAYPON 4CT	28	1562	16	98.9
STEOL 4N	86	1456	731	49.7
MIRANOL C2M	57	8192	78	99.0

C H E M T R I C

Table A-4   SCREENING PERFORMANCE OF UNIVERSAL'S CAB MEMBRANE

Solute	Permeation Rate (ml/hr)	Concentration Feed (ppm)	Permeate (ppm)	Rejection (%)
Deionized Water only	37			
Sodium Chloride	34	1502	17	98.9
Castile Soap	6	1340	Sample too small	
MIRANOL C2M	14	1776	44	97.5
HYAMINE 10-X	No data - leak developed			
VANCIDE BN	3	451	Sample too small	

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C H E M T R I C

Table A-5 SCREENING PERFORMANCE OF AMICON'S DIAFLO UM-05 MEMBRANE

| Solute | Permeation Rate (ml/hr) | Concentration | | Rejection (%) |
		Feed (ppm)	Permeate (ppm)	
Deionized Water Only	578			
Sodium Chloride	498	1502	1200	20.1
Castile Soap	126	1340	20	98.5
MIRANOL C2M	421	1776	579	67.5
HYAMINE 10-X	295	494	28	94.5
VANCIDE BN	295	451	12	97.3
HYAMINE 3500	324	553	137	75.2
PVP-Iodine 10	210	421	11	97.3
STEPANOL WA-100	Surfactant degraded membrane			
BIOTERGE AS-35CL	348	1324	311	76.6

C H E M T R I C

Table A-6 SCREENING PERFORMANCE OF DUPONT'S PERMASEP UNIT

(S/N 3598-139-30)

Solute	Permeation Rate (ml/hr)	Concentration Feed (ppm)	Permeate (ppm)	Rejection (%)
Deionized Water Only	280			
HYAMINE 3500	199	579	587	~ 0
Castile Soap	165	1521	427	68.1
BIOTERGE AS-35CL	141	1206	487	59.4
BIO SOFT EA-10	126	1460	606	51.6
MIRANOL C2M	184	1879	611	67.5
Sodium Chloride	178	1510	508	66.3
HYAMINE 10-X	157	499	300	39.9
STEPANOL WA-100	152	1359	995	26.8
BTC 2125	117	496	642	~ 0
MAYPON 4C	56	1451	605	58.3

C H E M T R I C

Table A-7 SCREENING PERFORMANCE OF DUPONT'S PERMASEP UNIT

(S/N 3598-139-29)

Solute	Permeation Rate (ml/hr)	Concentration Feed (ppm)	Permeate (ppm)	Rejection (%)
Deionized Water Only	213			
Sodium Chloride	210	1446	133	90.7
VANCIDE BN	182	464	22	95.3
PVP-Iodine 10	148	472	84	82.3
MIRANOL C2M	150	2600	443	83.0
HYAMINE 10-X	135	545	339	37.8

C H E M T R I C

Figure A-3, a plot of membrane flux versus time, illustrates the effect of pHisoHex on membrane performance; this test was performed to corroborate the previous results obtained by Aerojet-General. The run with pHisoHex and Eastman's RO-97 membrane was extended over 18 hours. Initially, the cell was fed deionized tap water for 5-1/2 hours to establish a calibration, and then the feed was changed to 532 ppm pHisoHex. After 15-1/2 hours, the feed was changed to deionized water to calibrate the cell. The decline in performance is attributed to Hexachlorophene in the pHisoHex formulation; Hexachlorophene deposits on the membrane surface and fouls it. Any preparation containing Hexachlorophene will give analogous results with all membranes - because Hexachlorophene is very insoluble.

Compatibility problems exist with organic solutes. Anionic materials can react with cationic materials to form sols or precipitates. The antiseptic characteristics of a germicide can be reduced with materials of the opposite type; i.e., anionic germicides can be inactivated by cationic materials, and cationic germicides can be inactivated by anionic materials. On the basis of compatibility, an amphoteric material, namely, Miranol, appears to be the most attractive surfactant for wash-water applications. It is compatible with anionic, cationic and nonionic agents in all proportions.

A.4.2.3 Conclusions & Recommendations

The following conclusions and recommendations were based upon the results obtained from the screening tests.

1. AMICON's Diaflo UM-05, an ultrafiltration membrane, is substantially more permeable than the other membranes tested; however, it has relatively low rejection capabilities, and it was found to be chemically degraded by sodium lauryl sulfate which is a principle ingredient of dentifrices.

2. The most permeable cellulose acetate membrane tested was Eastman's RO-97; therefore, it was selected for parametric testing. DuPont's Permasep unit was also selected for further study because it (1) uses a relatively inert isotropic polymer, (2) was found to effectively reject all solutes except benzalkonium chloride, and (3) is designed to contain a relatively large amount of membrane area in a small volume.

3. When cationic and anionic species are present in wash water, they form sols and/or precipitates which can "foul" the surface of an RO membrane; therefore, Miranol, an amphoteric surfactant, is recommended when wash water is to be renovated by RO.

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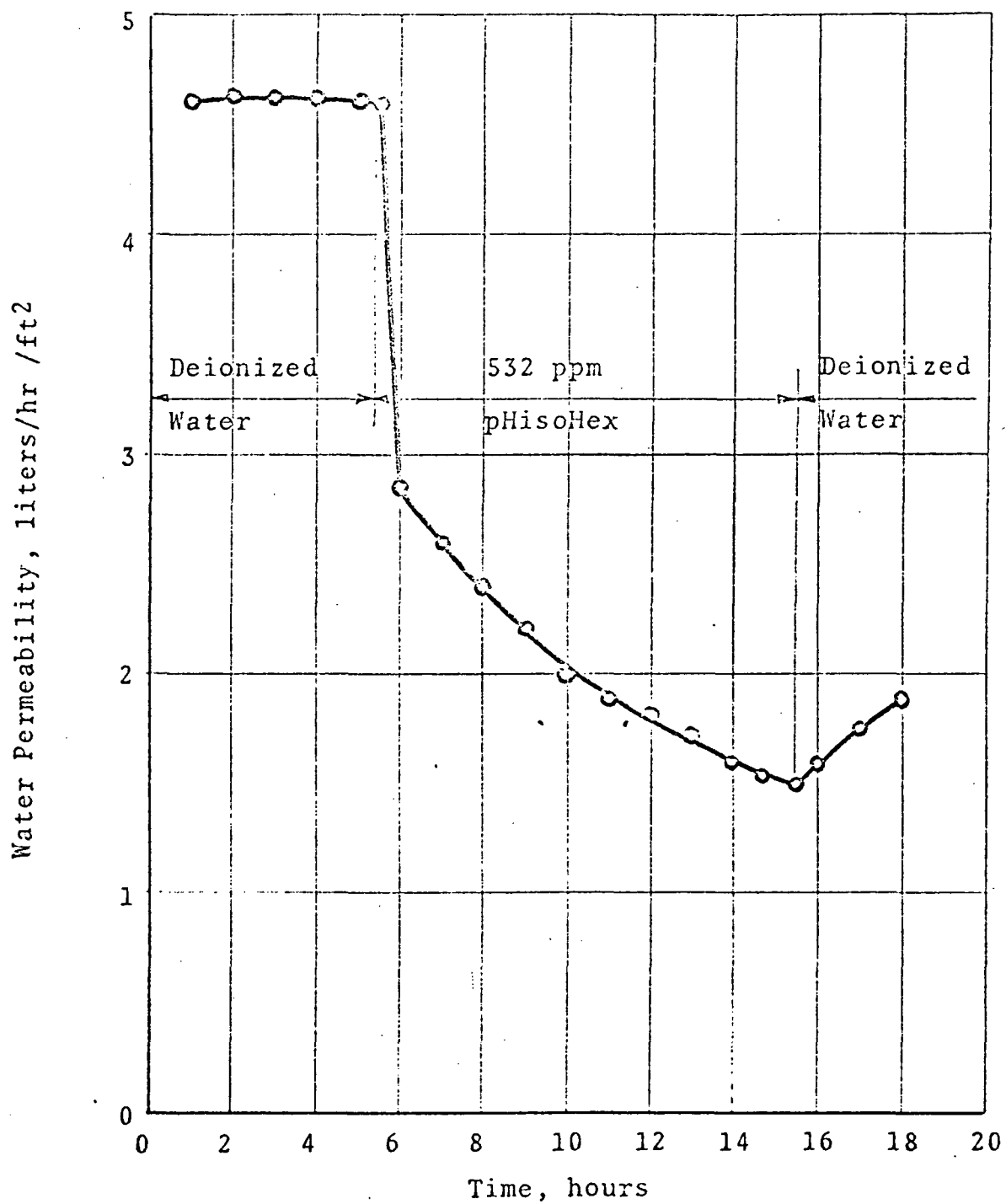
Figure A-3 EFFECT OF PHISOHEX ON PERMEABILITY

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C H E M T R I C

4. The best germicide, at least from the standpoint of membrane performance and compatibility, is Vancide BN. Iodophors chemically degrade membranes, while insoluble Hexachlorophene fouls the surface of membranes. Quartenary ammonium salts can be used - but they are not rejected as easily as Vancide BN.

A.4.3 Parametric Tests

In this task two selected membrane materials (Eastman's RO-97 and DuPont's Permasep) were subjected to detailed reverse osmosis experiments. The feed concentration, applied pressure (pumping pressure), circulation velocity, feed rate and/or recycle ratio were varied. Preliminary life time tests were also conducted.

A.4.3.1 Test Procedure

Three different samples of Eastman's RO-97 membrane (from the same lot) and one Permasep Unit (#3598-139-27) were used in this test series. The test apparatus described in Section A.4.1 was employed. Various concentrations of Miranol C2M-SF and NaCl were processed under varied hydrodynamic conditions.

The system was initially fed test solution for one hour during each run to establish an operating equilibrium. Brine and permeate streams were then collected to determine stream flow rates and their quality. The run was usually terminated when 500 ml of permeate was collected (the amount necessary to perform the analyses). Laminar flow (11 cm/sec for film materials) was obtained by feeding 0.6 gph with the hydraulically activated diaphragm pump. Turbulent flow (130 cm/sec for film materials) was attained by recycling 7.2 gph of the feed solution in the recyle loop with a modified Eastern Industries gear pump. Applied pressure was varied by changing the pressure charge within the gas dome of the back-pressure regulator.

A.4.3.2 Test Results

Table A-8 lists and sumarizes the operating characteristics of Sample A of Eastman's RO-97 membrane at various pressures and hydrodynamic conditions, while processing a feed containing approximately 1% Miranol and 0.1% NaCl (simulating process conditions at 90% water recovery). Table A-9 lists and summarizes the characteristics of Sample B of Eastman's RO-97 membrane at various pressures and hydrodynamic conditions while processing a feed containing approximately 1500 ppm Miranol and 150 ppm NaCl (simulating input feed conditions). Figure A-4 shows the observed flux characteristics of Samples A and B as a function of pressure.

CHEMTRIC

Hydrodynamic Conditions		Stream	Flow Rate	Stream Quality Characteristics				
(psi)	(cm/sec)		(ml/hr)	pH	Sp. Res. (Ω-cm)	T S (ppm)	NaCl (ppm)	Miranol (ppm)
600	11	Feed	2249	9.8	340	10034	969	9165
		Brine	2116	9.8	290	10638	965	9673
		Permeate	133	6.7	16000	386	331	55
600	130	Feed	2284	9.8	340	10034	969	9165
		Brine	2152	9.7	300	11089	967	10122
		Permeate	132	7.0	43000	115	127	- -
400	11	Feed	2336	9.8	300	9848	969	8879
		Brine	2240	9.7	289	10602	966	9636
		Permeate	96	9.2	1310	472	389	83
400	130	Feed	2295	9.6	310	9758	969	8789
		Brine	2200	9.6	280	10116	967	9149
		Permeate	95	8.7	1800	460	292	168
200	11	Feed	2361	9.8	330	10660	959	9701
		Brine	2317	9.8	290	10775	959	9816
		Permeate	44	9.1	760	997	750	247
200	130	Feed	2356	9.8	300	10584	1039	9545
		Brine	2316	9.8	300	11000	1016	10984
		Permeate	40	9.8	720	1063	890	173

C H E M²T R I C

Table A-9   LOW SOLIDS PERFORMANCE OF EASTMAN'S RO-97 MEMBRANE

Conditions (psi)	(cm/sec)	Stream	Rate (ml/hr)	pH	Stream Quality Characteristics Sp. Res. ($\Omega \cdot cm$)	T S (ppm)	NaCl (ppm)	Miranol (ppm)
600	11	Feed	2101	9.7	1900	1639	146	1493
		Brine	1982	9.5	2300	1696	149	1547
		Permeate	119	7.3	38000	14	9	5
600	130	Feed	2305	9.6	1700	1552	146	1406
		Brine	2190	9.6	1650	1595	144	1452
		Permeate	115	7.1	56000	3	7	0
400	11	Feed	2350	9.7	1700	1552	146	1406
		Brine	2270	9.6	1600	1690	162	1528
		Permeate	80	6.8	33000	20	20	0
400	130	Feed	2347	9.7	1750	1575	146	1429
		Brine	2274	9.7	1600	1633	155	1482
		Permeate	73	7.2	54000	8	7	1
200	11	Feed	2381	9.4	1700	1673	165	1483
		Brine	2340	9.4	1700	1707	183	1624
		Permeate	41	7.3	21000	55	23	32
200	130	Feed	2363	9.4	1700	1648	165	1483
		Brine	2324	9.3	1690	1651	188	1463
		Permeate	39	6.8	25000	13	18	---

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C H E M T R I C
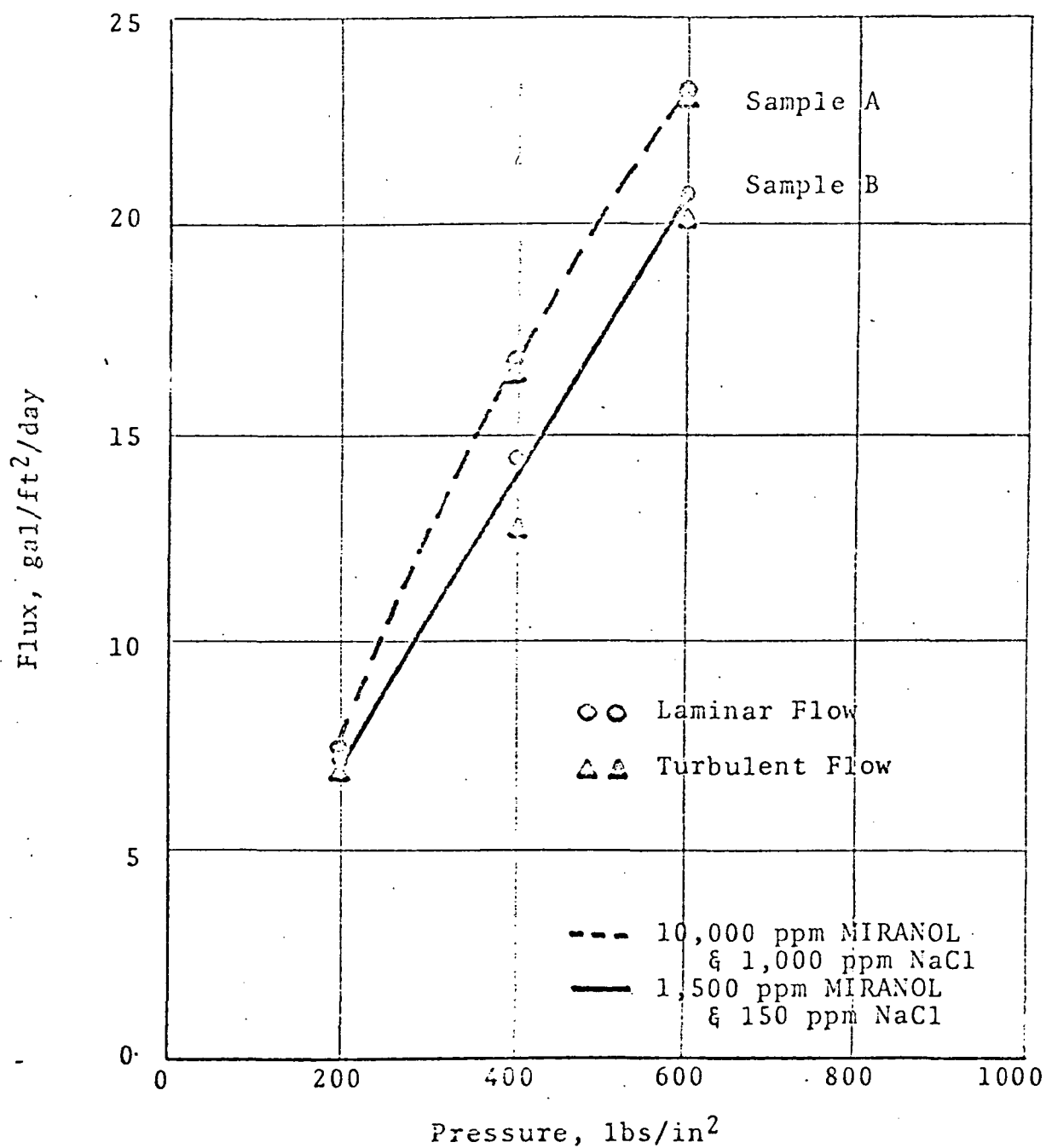
Figure A-4   PERFORMANCE OF EASTMAN'S RO-97 MEMBRANE

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Table A-10 lists and sumarizes the operating characteristics of Sample C of Eastman's RO-97 membrane while processing various concentrations of NaCl at 600 psi under various hydrodynamic conditions. Table A-11 lists and summarizes the operating characteristics of this membrane, while processing various concentrations of Miranol C2M and NaCl at 600 psi under various hydrodynamic conditions. Figure A-5 presents the flux characteristics of Sample C from the data in Tables A-10 and A-11 as a function of present simulated recovery at 600 psi.

Tables A-12 through A-17 list and summarize the operating characteristics of DuPont's Permasep Unit #3598-139-27 when processing various concentrations of Miranol and NaCl at various pressures and hydrodynamic conditions. Figure A-6 presents the flux characteristics of this unit as a function of solute concentrations at various hydrodynamic conditions.

A.4.3.3 Discussion of Results

The data presented in Figure A-4 is contradicting - i.e., the flux observed when processing test solutions approximately one order of magnitude greater in concentration, were higher than when processing dilute test solutions. The sample C tests were performed to determine whether membrane variations or some other phenomena caused the difference in performance between Samples A and B of Eastman's RO-97 membrane; salt, and then salt and Miranol were processed. As shown in Figure A-5, flux decreased as yield was forced to increase - however, performance continued to also decrease with time. The membrane was then recalibrated (terminal conditions) and found to be substantially less permeable then it was at the beginning of this test series. Examination of the membrane after termination of the test series showed no evidence of surface fouling. The only explanation for the nonrecoverable loss in flux appears to be "membrane compaction".

It is well established that modified membranes show decreasing water-permeation rates with time at fixed pressure,* as the results of long-term creep. The relatively large degradation observed after only 58 hours suggests that surfactants accelerate compaction effects. Cursory examination of another Eastman RO-97 membrane soaking in 1% Miranol, 0.1% NaCl and 0.1% Hyamine 3500 brine showed that the membrane characteristics changed during the soaking; the membranes appeared to be more pliable.

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*Merten, U., "Desalination by Reverse Osmosis", the M.I.T. Press, Cambridge, Massachusetts, 1966, p 145-150.

Table A-10 NaCl RETEST OF EASTMAN'S RO-97 MEMBRANE

Hydrodynamic Conditions (psi)	(cm/sec)	Stream	Flow Rate (ml/hr)	pH	Stream Quality Characteristics Specific Resistance (Ω-cm)	NaCl (ppm)
600	11	Feed	2301	5.7	1300000	0
		Brine	2164	6.1	360000	Deionized
		Permeate	137	5.9	1180000	Water)
600	11	Feed	2296	6.0	1700	330
		Brine	2166	6.2	1550	352
		Permeate	130	5.8	7300	63
600	130	Feed	2291	5.5	1630	330
		Brine	2176	5.9	1550	362
		Permeate	115	5.5	14000	37
600	11	Feed	2310	6.0	960	676
		Brine	2193	6.2	770	697
		Permeate	117	5.9	1800	332
600	130	Feed	2309	5.2	880	676
		Brine	2207	6.2	820	707
		Permeate	102	5.9	3720	86
600	11	Feed	2308	5.3	600	968
		Brine	2201	5.3	560	981
		Permeate	107	5.9	920	664
600	130	Feed	2302	5.7	600	968
		Brine	2205	6.0	590	1013
		Permeate	97	6.0	5000	118
600	11	Feed	2313	5.2	320	2065
		Brine	2208	5.5	280	2065
		Permeate	105	5.9	880	719
600	130	Feed	2323	5.2	300	2065
		Brine	2210	5.7	300	2069
		Permeate	113	5.8	1740	361

C H E M T R I C

Table A-11   NaCl & MIRANOL RETEST OF EASTMAN'S RO-97 MEMBRANE

Hydrodynamic Conditions (psi)	(cm/sec)	Stream	Flow Rate (ml/hr)	pH	Sp. Res. (Ω-cm)	(ppm)	(ppm)	Miranol (ppm)
600	11	Feed	2305	9.6	820	3701	347	3354
		Brine	2202	9.6	730	3888	362	3526
		Permeate	103	8.4	4890	116	97	19
600	130	Feed	2249	9.5	760	3809	347	3462
		Brine	2143	9.6	740	3963	359	3604
		Permeate	106	8.0	17000	31	27	4
600	11	Feed	2298	9.8	440	7750	664	7806
		Brine	2223	9.8	390	8199	693	7426
		Permeate	75	8.2	1800	520	278	242
600	130	Feed	2323	9.8	420	7625	693	6930
		Brine	2221	9.8	430	7775	716	7059
		Permeate	102	8.4	5900	97	85	12
600	11	Feed	2318	10.0	170	21180	1924	19250
		Brine	2255	9.9	150	22285	1932	20352
		Permeate	63	8.8	660	988	760	230
600	130	Feed	2318	9.9	160	22530	1840	20690
		Brine	2223	9.8	150	23310	1842	22468
		Permeate	95	9.4	640	1000	788	212
600	11	Feed	2312	5.9	2150000	0	0	0
		Brine	2226	5.9	1280000	(Deionized Water)		
		Permeate	86	7.0	440000			
600	130	Feed	2290	5.9	1200000	0	0	0
		Brine	2205	6.2	800000	(Deionized Water)		
		Permeate	85	7.0	580000			

C H E M T R I C

Table A-12 RAW FEED TEST OF DU PONT'S PERMASEP UNIT

Hydrodynamic Conditions (psi)	(gpm)	Stream	Flow Rate (ml/hr)	Stream Quality Characteristics				
				pH	Sp. Res. (Ω-cm)	T S (ppm)	NaCl (ppm)	Miranol (ppm)
600	0	Feed	2306	9.15	1720	1645	152	1510
		Brine	2118	9.15	1540	1765	167	1598
		Permeate	188	8.8	8540	150	47	103
650	0.12	Feed	2442	9.5	1720	1645	152	1510
		Brine	2263	9.4	1600	1693	164	1529
		Permeate	178	8.8	8400	220	49	171
400	0	Feed	2341	9.3	2480	840	150	690
		Brine	2193	9.3	2220	906	155	751
		Permeate	148	9.0	6700	167	66	101
450	0.12	Feed	2292	9.3	2380	840	150	690
		Brine	2133	9.1	2300	928	159	770
		Permeate	159	8.6	6610	172	65	107
200	0	Feed	2386	9.7	1800	1662	152	1510
		Brine	2302	9.6	1640	1702	158	1547
		Permeate	84	9.3	4650	299	92	207
250	0.12	Feed	2329	9.5	1720	1661	152	1510
		Brine	2237	9.5	1700	1674	163	1511
		Permeate	92	9.0	4720	245	96	149

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C H E M T R I C

Table A-13 50% YIELD TEST OF DUPONT'S PERMASEP UNIT

Hydrodynamic Conditions (psi)	(gpm)	Stream	Flow Rate (ml/hr)	pH	Sp. Res. (Ω-cm)	T S (ppm)	NaCl (ppm)	Miranol (ppm)
600	0	Feed	2303	9.8	1120	2681	206	2475
		Brine	2114	9.6	980	2896	231	2665
		Permeate	189	9.6	5350	260	62	198
650	0.12	Feed	2293	9.8	1120	2681	206	2475
		Brine	2058	9.6	910	2858	233	2625
		Permeate	235	9.6	4900	244	66	178
400	0	Feed	2348	9.8	1120	2681	206	2475
		Brine	2193	9.8	1000	2715	223	2492
		Permeate	155	9.6	3900	376	90	286
450	0.12	Feed	2323	9.8	1120	2681	206	2475
		Brine	2157	9.5	1000	2880	223	2657
		Permeate	166	9.3	3800	315	92	223
200	0	Feed	2380	9.9	1120	2805	208	2597
		Brine	2294	9.8	980	2762	226	2536
		Permeate	86	9.3	2700	465	125	340
250	0.12	Feed	2346	9.9	1140	2805	208	2597
		Brine	2236	9.8	1100	2794	210	2584
		Permeate	110	9.6	3100	455	130	325

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Table A-14 70% YIELD TEST OF DuPONT'S PERMASEP UNIT

Hydrodynamic Conditions			Flow Rate	Stream Quality Characteristics				
(psi)	(gpm)	Stream	(ml/hr)	pH	Sp. Res. (Ω·cm)	TS (ppm)	NaCl (ppm)	Miranol (ppm)
600	0	Feed	2308	9.9	820	3743	287	3456
		Brine	2110	9.8	720	4055	316	3739
		Permeate	198	9.5	3450	341	102	239
650	0.12	Feed	2264	9.9	820	3743	287	3456
		Brine	2040	9.3	670	4120	335	3785
		Permeate	224	9.6	3600	355	87	368
400	0	Feed	2343	9.9	820	3743	287	3456
		Brine	2187	9.7	740	3939	317	3622
		Permeate	156	9.2	2900	428	128	300
450	0.12	Feed	2343	9.9	820	3743	287	3456
		Brine	2187	9.7	700	4058	322	3736
		Permeate	175	9.4	2800	452	131	321
200	0	Feed	2381	9.8	800	3683	286	3397
		Brine	2294	9.6	760	3829	302	3478
		Permeate	87	9.2	2000	596	196	412
250	0.12	Feed	2337	9.8	800	3683	286	3397
		Brine	2240	9.6	760	3829	299	3530
		Permeate	97	9.2	2000	596	199	397

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Table A-15   85% YIELD TEST OF DuPONT'S PERMASEP UNIT

Hydrodynamic Conditions		Stream	Flow Rate (ml/hr)	Stream Quality Characteristics				
(psi)	(gpm)			pH	Sp. Res. (Ω-cm)	T S (ppm)	NaCl (ppm)	Miranol (ppm)
600	0	Feed	2313	9.9	520	6140	445	5695
		Brine	2138	10.0	480	6406	452	6154
		Permeate	175	9.6	2400	430	168	262
650	0.12	Feed	2289	9.9	520	6140	445	5695
		Brine	2068	9.9	460	6540	457	6083
		Permeate	221	9.7	2180	532	160	372
400	0	Feed	2340	9.9	520	6140	445	5695
		Brine	2186	9.8	480	6238	454	5829
		Permeate	154	9.6	1400	705	231	474
450	0.12	Feed	2330	9.9	520	6140	445	5695
		Brine	2160	9.9	480	6368	452	5916
		Permeate	170	9.6	1800	642	215	427
200	0	Feed	2360	9.9	540	5825	434	5391
		Brine	2274	9.8	560	5980	446	5534
		Permeate	86	9.4	1200	932	342	590
250	0.12	Feed	2350	9.9	540	5825	434	5391
		Brine	2232	9.8	490	6030	442	5588
		Permeate	118	9.5	1300	531	318	213

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Table A-16   90% YIELD TEST OF DuPONT'S PERMASEP UNIT

Hydrodynamic Conditions (psi)	(gpm)	Stream	Flow Rate (ml/hr)	pH	Sp. Res. (Ω-cm)	T S (ppm)	NaCl (ppm)	Miranol (ppm)
600	0	Feed	2367	10.1	300	11007	954	10005
		Brine	2301	10.0	270	9036	992	8044
		Permeate	166	9.7	1050	904	475	429
650	0.12	Feed	2305	10.0	300	10692	954	9738
		Brine	2123	10.0	280	11418	976	10418
		Permeate	182	9.4	1020	878	515	363
400	0	Feed	2346	10.0	300	10604	954	9650
		Brine	2207	10.0	290	11240	972	10268
		Permeate	139	9.5	790	1131	640	491
450	0.12	Feed	2369	10.0	300	10710	954	9756
		Brine	2232	10.0	290	12548	981	11567
		Permeate	137	9.5	780	1126	637	489
200	0	Feed	2394	10.1	330	12010	972	11038
		Brine	2313	10.0	300	11500	994	10586
		Permeate	81	9.4	580	1615	890	725
250	0.12	Feed	2370	10.1	300	11008	972	10003
		Brine	2286	10.1	290	11400	997	10403
		Permeate	84	9.5	610	1521	846	675

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Table A-17 95% YIELD TEST OF DuPONT'S PERMASEP UNIT

| Hydrodynamic Conditions (psi)	(gpm)	Stream	Flow Rate (ml/hr)	pH	SP. Res. (Ω-cm)	T S (ppm)	NaCl (ppm)	Miranol (ppm)
600	0	Feed	2323	10.1	170	22430	1531	20900
		Brine	2139	10.1	150	25462	1489	23973
		Permeate	184	9.8	510	2640	916	1724
650	0.12	Feed	2310	10.1	170	22430	1531	20900
		Brine	2093	10.1	150	25064	1489	23575
		Permeate	217	9.7	500	2108	914	1194
400	0	Feed	2351	10.0	160	22430	1531	20900
		Brine	2190	9.9	150	22520	1590	20930
		Permeate	161	9.7	360	2256	1157	1099
450	0.12	Feed	2315	10.0	160	22430	1531	20900
		Brine	2130	10.0	150	22701	1590	21111
		Permeate	185	9.6	380	2185	1115	1070
200	0	Feed	2370	9.9	180	21823	1539	20280
		Brine	2286	10.0	150	23290	1607	21680
		Permeate	84	9.4	270	3180	1410	1688
250	0.12	Feed	2309	9.9	180	21823	1539	20280
		Brine	2204	10.0	150	23480	1588	21890
		Permeate	105	9.6	320	2930	1469	1462

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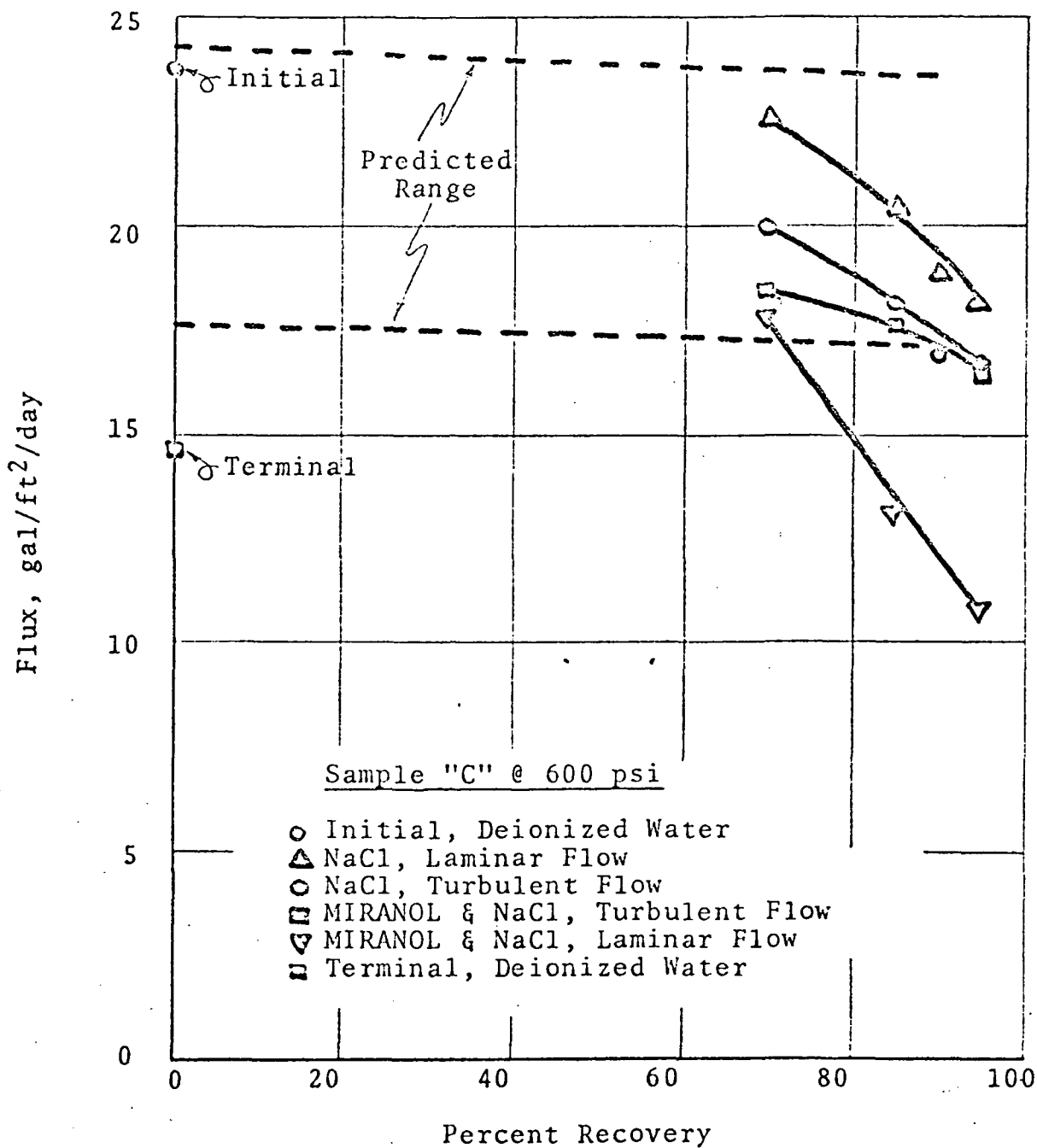
Figure A-5   RESTEST OF EASTMAN'S RO-97 MEMBRANE

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The results presented in Tables A-8, 9, 10 and 11, and Figures A-4 and A-5 show that Eastman's RO-97 membrane, and perhaps other modified or Loeb-type cellulose acetate membranes, are not suitable for wash water recovery. The Loeb-type or modified membrane consists of two rather well-defined regions; one an essentially non-porous surface layer and the other a highly porous supporting structure which comprises the bulk of the membrane. The thin surface layer is estimated to be a few hundred Angstroms deep. Three possible explanations of the changes in membrane properties with compaction are possible.* First, the water permeability may be time or pressure dependent. Second, it is possible that viscous resistance to flow in the porous substructure increases with time or pressure to the point where it is a substantial fraction of the total resistance to flow in the membrane. Third, it is conceivable that the salt-rejecting surface layer of the membrane grows in thickness due to plastic flow or creep.

The results obtained with the Permasep unit (Tables A-12 through A-17 and Figure A-6) show that (1) hydraulic permeability and solute retention increases with increasing applied pressure, and (2) at constant pressure hydraulic permeability decreases with increasing concentration. The data shows that the initial and terminal calibration with deionized water were within 10%; the terminal flux observed, in fact, was higher in two cases. No loss in flux was observed after 230 hours of operation under pressure. During these tests, the Permasep unit had been in contact with Miranol and NaCl brine at a pH of 9.5 to 10.0 for 55 days.

The data listed in Tables A-8 through A-17 shows that recycling of the brine to obtain higher velocities did not significantly improve performance, except in the case of electrolytes (NaCl). The explanation for this result is that surfactants, like Miranol, have molecular weights about an order of magnitude greater than NaCl; consequently, they exhibit lower osmotic pressures even when they are present in a laminar boundary layer on the membrane.

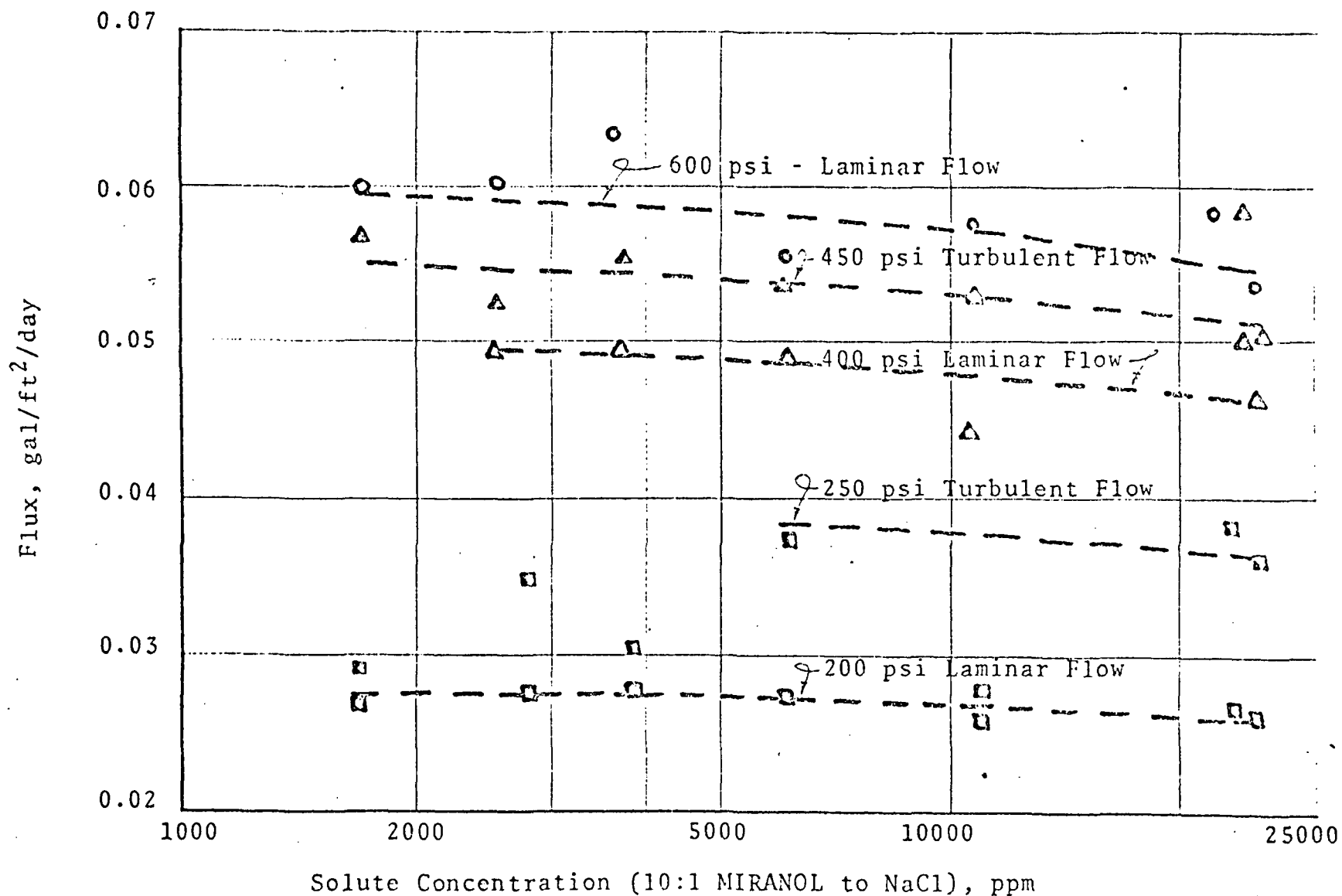* Merten, U., op.cit.

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Figure A-6   PERFORMANCE OF PERMASEP UNIT No. 3598-139-27

A.4.3.3 Conclusions & Recommendations

The following conclusions and recommendations are based upon the results obtained from the parametric tests.

A. Eastman's RO-97 membrane experiences accelerated compaction when processing Miranol C2M; therefore, it cannot be recommended for use in an integrated water and waste management system.

B. DuPont's B-5 Permasep material does not experience any degradation after 230 hours of pressure operation and 55 days of soaking in Miranol and NaCl brine; therefore, it is the recommended material for renovating used wash water.

C. Recycling brine to increase turbulence does not provide any significant gains in permeability and/or permeate quality; therefore, single pass operation is recommended for an integrated water and waste management system.

SOLIDS DRYING INVESTIGATIONS

B.1 Background

An important objective of any water recovery device is to extract as much of the available water in a waste liquid as is possible. The greatest possible yield would, of course, reduce the waste liquid to a dry powder or, in the case of urine waste, a heavy oil.

Two major difficulties arise in maximizing yield - namely, boiling point excursion, and evaporator or heat transfer surface fouling. As water is extracted from a waste liquid, the dissolved solids increase in concentration, thereby elevating the boiling point of the liquid; for urine slurries, a 40°F excursion occurs through the 5 to 90% solids concentration range.* Accumulation of a layer of solid material on the evaporator surfaces retards heat flow, thereby increasing energy demand or if energy is limited, lowering distillation rate.

The compression distillation unit developed by The Marquardt Corporation (TMC) uses mechanical wipers to clean the primary evaporator and to move the liquor/slurry over a "dam" into an electrically heated solids drying compartment within the still. The wipers along with their control circuitry introduces enormous complexity. The self-contained solids dryer, because of its limited capacity, precludes continuous operation and necessitates partial disassembly of the still to manually remove waste solids. In tests with these distillation units,** the wipers failed repeatedly and yields of only 50 to 70% were realized. Higher yields were not possible because vapor evolved from the solids condensed at locations where droplets could be "centrifuged" back into the solids dryer.

An alternate approach developed previously by CHEMTRIC personnel, for dealing with the fundamental problems, consisted of (1) chemical pretreatment of the waste, (2) recirculation of evaporator liquor at a rate several times greater than the evaporator rate, and (3) limiting water yield to value near 90%. The chemical pretreatment of the waste in combination with recircu-

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*Putnam, D. F., Chemical and Physical Properties of Human Urine Concentrates, NASA CR-66612, April, 1968.

**Nuccio, P. P., et. al., Refurbishment & Testing of the Integrated Waste Management System, AMGLO Final Report 3080, Contract NAS9-9014, October 1969.

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lation prevents permanent evaporator fouling. Yield limitation limits the evaporator liquor solids concentration;* by limiting solids concentration, a narrow boiling point range is maintained and the probability of evaporator fouling is reduced.

The above features were incorporated in a compression distillation unit developed and tested for the Air Force.** In tests of this unit, an overall yield of 86% was obtained along with an average specific energy value of 45.5 watt-hours/# water. The solids concentration of the evaporator liquor did not exceed 35% in these tests; normally, the concentration remained near 30% solids.

The disadvantage in the above approach is the lower than desirable water yield and the fact that concentrated waste has to be stored and/or disposed of. Development of a solids dryer, that was adaptable to a compression distillation unit, was undertaken to maximize yield and to provide a means of managing concentrated waste liquor.

The concept chosen as the starting point consisted of an externally located disposable boiler which utilizes capillary media to restrain the waste liquor during drying in a weightless state. Water vapor generated in the drying process would be vented to the evaporator of the distillation unit via a passive condenser. Electric heaters would provide the driving force for evaporation. The dryer would interface with the recycle loop, evaporator and electrical power source. A target useful life of 30 days (120 man-days) was arbitrarily selected as a reasonable period between dryer changes.

The test program summarized in the following sections was undertaken to (1) evaluate available capillary media for compatibility with the subject application, (2) determine the waste solids capacity of candidate media, and (3) acquire data and experience for the design of a full-scale dryer.

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* Yield or liquor solids concentration can be limited in a continuous process at any target value by bleeding-off or dumping evaporator liquor at a rate such that the solids quantity dumped equals the quantity of fresh solids introduced.

**Nuccio, P. P., and Jasionowski, W. J., Automatic Water Recovery System, AMRL-TR-67-155, March 1968.

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The test effort was divided into static and dynamic tests of capillary media, and long-term solids loading tests; the solids loading tests were conducted on one capillary medium selected on the basis of the preceeding evaluation tests.

Evaluation of various capillary media was deemed necessary since the subject application differed significantly from existing uses of such media. In this application, the medium is heated directly and evaporation takes place throughout the bulk of the medium, thereby adding the requirement of vapor permeability to liquid retention capability. The medium should also possess good horizontal as well as vertical wicking capability to maximize volume utilization and to avoid elaborate waste liquid input manifolds. Compatibility with pretreatment chemicals also required evaluation.

The initial test plan called for testing a broad variety of capillary media. However, very few materials could be obtained in suitable thickness in sufficient time for inclusion in the evaluation. Two types of felt (namely, wool and polypropylene) along with several types of sponge were procured for evaluation.

B.2 Static Tests of Capillary Media

The initial tests consisted of measuring the static liquid retention capability of the candidate media types. These tests were conducted as follows.

Small blocks (2" x 2" x 0.5") of the capillary media were first weighed and then soaked in water, urine, and urine brine. The wet blocks were then placed on a coarse screen, narrow side down, and allowed to drain for 30 minutes. The weight gain was taken as the static liquid capacity of the given media. Distilled water and freshly collected urine containing pretreatment chemical and urine brine were used as the test liquids. Urine brine was prepared from freshly collected urine, containing pretreatment chemical, by distillation in a laboratory-type, vacuum distillation assembly at 80°F; the specific gravity of the brine used in all tests was maintained within the range of 1.130 and 1.140.

The results of these tests are presented in Table B-1 on the next page. Liquid retention capability is expressed as the ratio of the liquid retained to the weight of the test piece. A weight ratio was used as the measure of retention rather than volume ratio, because of the near impossibility of cutting a dimensionally precise test piece from such soft and pliable materials. The data does show that the sponge materials, exclusive of coarse-grade urethanes, are capable of substantially greater liquid retention than the felt materials. On the basis of these tests, the 10 and 20 ppi urethanes were removed from further consideration.

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Table B-1 STATIC LIQUID RETENTION BY CAPILLARY MEDIA

Type	Grade	Approximate Density gms/cc	Liquid Retention(gm/gm) Water Media	Urine Media	Brine Media
Urethane Sponge	10 ppi*	0.033	1.1	1.1	1.1
Urethane Sponge	20 ppi	0.027	5.1	2.0	2.2
Urethane Sponge	30 ppi	0.026	8.5	4.2	5.6
Urethane Sponge	60 ppi	0.030	22.2	11.1	11.1
Urethane Sponge	80 ppi	0.030	26.2	21.1	23.4
Cellulose Sponge	Fine	0.033	19.8	17.8	18.7
Cellulose Sponge	Coarse	0.077	14.3	13.1	14.1
Cellulose/Nylon Sponge	Coarse	0.076	19.9	11.1	11.1
Wool Felt	Fine	0.280	3.9	2.6	2.7
Polypropylene Felt	Fine	0.265	3.8	3.0	3.0

* = pores per inch.

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B.3 Dynamic Tests of Capillary Media

The objective of these tests was to evaluate the liquid retention capability of candidate media under simulated use conditions. A miniature dryer was fabricated to permit these evaluations.

The construction of the dryer is illustrated in the figure on the next page. The dryer is a 4" diameter cylinder which could be adjusted by inserting various lengths of acrylic tubing. The majority of the tests were conducted with a 2" length of tubing; this size dryer could accommodate 25 in^3 of capillary media. The heater plate provided 12.6 in^2 of heat transfer area. Sizing of this dryer was somewhat arbitrary and represents a compromise between the need to base the evaluations on adequately sized pieces of capillary media without consuming excessive quantities of urine liquor; urine liquor preparation entails substantial logistics problems since approximately 10 liters of urine had to be collected and distilled to obtain one liter of urine liquor.

The dryer was incorporated into a test set-up which provided, (1) urine liquor input control, (2) heater power control and readout, (3) continuous temperature readout, (4) dryer pressure control and readout, (5) vapor condensation and collection, and (6) evaluation. The dryer was oriented in all tests such that gravity aided leakage of liquor into the vapor outlet line; this line was fabricated from pyrex tubing so that direct observation of any leakage could be made. The liquor retention capacity of a media was defined as that quantity of liquor that could be retained without leakage of liquor into vapor outlet.

B.3.1 Batch Tests

Batch-type dynamic tests of liquid retention capacity were conducted as follows, using 2" x 4" diameter cylindrical slabs of candidate capillary media. The media test piece was saturated with urine liquor while in place in the dryer. The dryer cover was then put in place and the pressure reduced gradually to 25 torr; this pressure was selected as being the minimum evaporator pressure that will be encountered in normal operation of a distillation unit. The latent heat content of the dryer, capillary medium, and urine liquor was sufficient to promote boiling for a brief period. Brine liquor in excess of the medium holding capacity drained into the vapor line and into an ambient temperature trap. After draining, the trap contents were weighed. The difference between the liquor input and trap contents was taken as the urine liquor holding capacity of that medium. The dryer heater was then turned on and maintained at a sufficient power level to maintain a 140°F heater temperature; this temperature level provided a 20°F margin over and above the
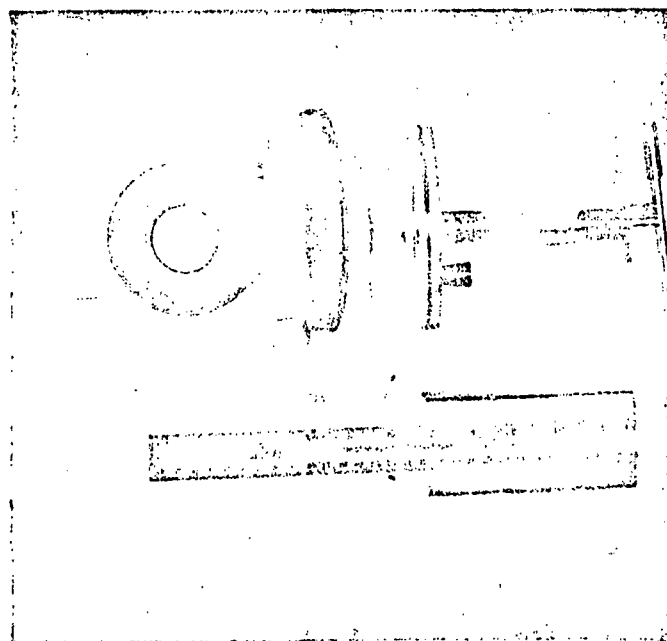
SHOWN, FROM LEFT-TO-RIGHT

· ELECTRIC HEATER

· METALIC BASE PLATE
  WITH VAPOR OUTLET
  HOLE ON CENTER

· ACRYLIC CYLINDER

· ACRYLIC COVER
  WITH INLET TUBE
  AND FEED TEE

Figure B-1   EXPERIMENTAL SOLIDS DRYER

estimated temperature necessary to maintain the saturation pressure of 90% solids liquor at 25 torr. After 24 hours of exposure to these conditions, the media test pieces were removed and weighed to determine the degree of dryness achieved.

The above test sequence provided a severe challenge to the capillary properties of any medium since gravity and a flowing vapor stream acted synergistically to promote leakage. The dryout sequence was pursued as a means of evaluating the combined impact of key media properties - namely, the effect of thermal conductivity and vapor permeability on the drying process.

The results of the batch tests are presented in the table following this paragraph. As shown, the liquor retention value for the cellulose/nylon sponge is substantially higher than the other candidate media; this high value was verified by a repeat test. The drying rate values are useful only for comparison between media types within the limits of the test procedure which did not provide regulation of the mass of the wetted test specimens, nor the total quantity of liquor available for evaporation.

Table B-2   DYNAMIC LIQUID RETENTION AND DRYING RATE

Media Description		Test Results	
		Liquor Retention Wt.Liquid/	Drying Rate
Type	Grade	Wt.Media	gms/hr
Cellulose/Nylon Sponge	Coarse	6.8	4.0
Cellulose Sponge	Coarse	3.8	3.0
Cellulose Sponge	Fine	3.4	3.5
Urethane Sponge	80 ppi*	2.8	1.0
Polyproplylene Felt	Fine	0.9	9.0
Wool Felt	Fine	0.8	8.0

*ppi = pores per inch

The above results favor the selection of cellulose/nylon sponge as the best medium for this application. This material is marketed under the Tradename NYLONGE and is intended for housekeeping uses. Nylonge is coarse-grade regenerated cellulose sponge, containing randomly distributed bundles of nylon fiber; the nylon fibers act as strengtheners to increase the resistance of the sponge to mechanical wear and tear. The manufacturer claims that the material is resistant to microbial attack and will withstand extended exposure to 100°C steam. The material is flammable as are all of the media tested.

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The selection of Nylonge is based primarily on liquor retention. The substantially better drying rate offered by the felt could be utilized advantageously, but at the expense of dryer weight. The felts are, at the least, 5 times more dense than the sponge materials and Nylonge can contain almost 7 times the amount of liquor. Based on these considerations, the mass of the felt media needed to contain a given quantity of liquor would be 35 times greater than the mass of Nylonge needed to contain the same amount of liquor. It is doubtful that any other weight penalty, attributable to Nylonge, could be sufficiently large to offset this basic weight disadvantage of the felt material.

B.3.2 Continuous Liquor Feed Tests

These tests were undertaken to evaluate the behavior of the media under conditions that closely approximated actual use conditions. Refinement of the retention capability of the media was a major goal along with acquisition of data and experience for designing a full-scale dryer.

A total of four tests were run with 2" x 4" diameter slabs of Nylonge. The mass of the test pieces varied between 22.5 and 25.6 grams. This mass variation is due to the near impossibility of cutting a dimensionally precise slab from such soft material; also, the dry Nylonge is readily compacted during cutting. Urine liquor obtained as in the previous tests was used in all four of the continuous feed tests. Condenser pressure was maintained at 25 $\pm$ 2 torr for all tests. The heat transfer plate temperature was maintained at 140 $\pm$ 2°F during the first two tests and 160 $\pm$ 5°F during the last two tests. The urine liquor feed rate varied with each test. The first two tests were run with automatic feeding, which permitted continuous 24-hour operation. Breakthrough of urine liquor into the ambient temperature trap was used as the criterion for determining the retention capacity of the media. The following describes each test and presents the data obtained.

Test No. 1

The first test was preliminary in nature and was devoted to determining the feed rate best suited for the experimental dryer. The dryer was fed 75 ml of urine liquor over a two-hour period, as a precharge to accelerate urine liquor accumulation. After precharging, urine brine was fed at an initial rate of 20 ml/hour. Breakthrough occurred after 2.75 hours. After a 1-hour "dry-out" period, the brine was fed at 10 ml/hour; breakthrough occurred in 4 hours. A feed rate of 7.5 ml/hour was then used after a 1-hour "dry-out" period. After 7 hours at 7.5 ml/hour, the test was terminated because the slow accumulation of condensate suggested that the vapor outlet (1/4" diameter)

had become plugged. A total of 257 grams (232 ml) of liquor had been fed up to this point. Upon disassembly, dried solids were found to have penetrated the vapor manifold and had occluded the outlet port.

The test indicates that the proper feed rate is very near to the previously determined drying rate. Because of the multiple breakthroughs that occurred, conclusions concerning capacity cannot be made. The test does point out a consequence of liquor leakage, namely, vapor line plugging.

Test No. 2

The test was initiated by precharging the dryer with 75 ml of liquor and then feeding at 7.5 ml/hour. This feed rate was maintained for 45 hours, at which time liquor breakthrough occurred. A total of 460 gms (415 ml) had been fed up to this time. The wet media test piece contained 193.9 gms of urine liquor at breakthrough. The dry solids mass in the test piece amounted to 101.0 gms after drying under a hard vacuum at 140°F for 24 hours. The solids concentration in the dryer liquor at breakthrough amounted to 52% as calculated from the above data. Inspection of the sectioned test piece revealed that most of the urine solids were confined to a 1/4" - 1/2" thick layer adjacent to the heat transfer plate.

The relatively low solids content of the liquor in the test piece at breakthrough may have been due to feeding at too high a rate or to a greater than anticipated boiling point excursion. The literature indicates that the vapor pressure of a 52% solids liquor would be 117 torr, which is well above the 25 torr dryer pressure.

Test No. 3

Two changes were made in the test conditions. First, an automatic feed controller was used; this device permitted 24-hour operation of the dryer. Secondly, the plate temperature was maintained at 160°F, instead of the 140°F previously used. The feed rate remained the same.

The test was initiated by precharging the dryer with 75 ml urine brine over a 1-hour period. After precharging, a feed rate of 7.5 ml/hour was used. Breakthrough occurred after 558 gms (503 ml) had been fed to the dryer and after 67 hours of operation. After a 4-hour "dry-out" period, urine was fed at the reduced rate of 5.0 ml/hour. The test was allowed to run over the weekend. Breakthrough occurred during an unattended period; consequently, this second breakthrough incident cannot be fixed accurately. Breakthrough is estimated to have occurred after a total of 802 gms (723 ml) had been fed and 115 hours of operating time had elapsed. The total input, since feeding continued

beyond the breakthrough point, was 959 gms (864 ml). The test piece contained 236 gms of liquor at the end of the test; the dry solids content was found to be 156.6 gms after drying under a hard vacuum at 160°F. The apparent liquor solids concentration was 66%.

Significant improvement in liquor retention and liquor solids concentration were realized. The improvement may be due to the increased operating temperature, but the test sequence may have also contributed to the apparent improvement.

Test No. 4

The feeding sequence was altered in this test. No precharge of urine brine was used and the feed rate of 5.0 ml was maintained constant from test initiation up to the breakthrough point. All other conditions remained the same.

Breakthrough occurred after 112.5 hours and after a total of 625 gms (563 ml) had been fed to the dryer. The test piece contained 239 gms of liquor at breakthrough; the dry solids content amounted to 172.8 gms. The final calculated liquor solids concentration amounted to 72%. The dry solids were distributed in the test piece in much of the same way as in test #3.

The amount of liquor retained in this test was very similar to that retained in the previous test. The final liquor solids concentration did increase by 6%. This small increase may be due to a closer match between feed rate and drying rate.

Some additional observations not included in any of the above test descriptions are included here since they apply to all the above tests. In each case, the wet Nylonge specimens, when first removed from the dryer, exhibited a "dryness" gradient, that is, the test piece was driest at the specimen end interfacing with the heater plate and wettest at the liquor input end. This "dryness" gradient also correlated with an apparent solids concentration gradient as evidenced by the intensity of the color imparted to the test specimen by the urine liquor. A temperature gradient as measured by thermocouples matched the above gradients in direction. However, this temperature gradient did vary considerably during each test and appeared to be influenced by liquor feed rate.

B.4 Conclusions & Recommendations

The test data and observations suggest that drying was limited to a narrow zone adjacent to the heater plate. The distance between the heater plate and the outer boundary of this zone is determined most directly by heater plate temperature. At 160°F, this distance is approximately one inch, while at

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140°F the distance is 1/4 to 1/2 inch. The 160°F plate tempera-
ture induced a temperature gradient with an average temperature
sufficiently high to permit drying to a 72% solids level. It
is concluded that the low thermal conductivity of the medium ac-
counted for the performance realized rather than a marked devia-
tion from the estimated boiling point excursion.

Breakthrough appears to have occurred because the volume of
the liquor in the active drying zone exceeded the void volume of
the Nylonge medium; this is explained as follows.

1. The active drying zone was approximately a one-inch
   thick layer equivalent to 1/2 the total dryer volume,
   or 12.5 in^3.

2. The experimentally determined void volume of Nylonge is
   64% of the total; the void volume of the active drying
   zone would be 8 in^3.

3. The 239 grams of 72% solids liquor retained in the spec-
   imen in test #4 would amount to 10.9 in^3 as determined
   from published values relating urine solids concentra-
   tion with specific gravity.

The main source of error in the above is the estimate of the
volume of the active layer since the exact volume is difficult to
measure. The 8 in^3 is most probably low. Leakage occurs when
liquor fills the media voids at the vapor outlet end, thereby
blocking vapor flow. Steam pressure, very low to be sure, de-
velops behind the liquor slug and blows it into the vapor line.

The following are established as guidelines for designing
a full-scale solids dryer.

1. The maximum operating temperature should not exceed
   160°F since gross thermal decomposition of urine con-
   stituents (e.g., urea) can occur above this temperature.

2. Capillary media volume requirement can be calculated
   from the void volume (64%) and the estimated volume of
   70 to 75% liquor that will accumulate during the desir-
   ed useful life of the dryer.

3. The capillary medium should not extend beyond one inch
   from the heaters or heat transfer plates.

4. The estimated water yield obtainable with a solids dry-
   er configured according to the above, in combination
   with compression distillation units, should be 100%
   less 1/3 x the percentage of dissolved solids in the
   waste water.

C H E M T R I C

FINAL REVERSE OSMOSIS INVESTIGATIONS

The final RO tests were divided into three tasks; namely, (1) Static Tests, (2) Filtration Tests, and (3) Compatibility Tests. The Static Tests entailed determination of prefiltration requirements of selected detergents and germicides, and the most promising combination of selected detergents and germicides for application. The Filtration Tests determined the turbidity of used wash water (i.e., shower water, face and hands water, clothes wash water, and dish wash water) before and after staged filtration. The Compatibility Tests (combination static storage and performance) determined the effects of the selected cleansing agents on the RO membranes of two candidate units; namely, DuPont's B9 Permasep Permeator and Westinghouse's 4 x 4 Basic Module.

C.1 Static Tests

Nine (9) candidate cleansing agent ingredients were tested for turbidity, miscibility and solubility at various concentrations that simulated loop processing conditions. Subsequently staged membrane filtrations were performed to determine the requirements to achieve turbidities of 1 and 0.1 JTU (Jackson Turbidity Units); 0.1 JTU's is recommended for DuPont's B9 unit.

C.1.1 Materials and Concentrations

The preselected detergents and germicides tested were as follows:

Detergents

Miranol C2M: Dicarboxylic coconut derived diethanolamine salt, colored liquid (Miranol Chemical Company)

Miranol JEM: Specifically designed for use as a wetting agent, colored liquid (Miranol Chemical Company)

Neutrogena Bar Soap: Mild, hypo-allergenic and bio-degradable bar soap (Neutrogena Corporation)

Neutrogena Rainbath Body Soap Gel: Mild body liquid soap gel for bath or shower - Tween 20, super amide, propylene glycol, sodium lauryl di-ether sulfate, methyl and propyl, parabens (Neutrogena Corporation)

Ivory Bar Soap: Mild bar soap (Procter & Gamble)

Tegobetaine-C: A fatty amidoalkyl betaine derived from coconut fatty acids, yellow-colored liquid (Goldschmidt Chemical)

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Germicides

Vancide BN: Sodium bithionolate, fine crystalline powder
(R. T. Vanderbilt Co.)

Vancide BN Solution: Sodium bithionolate solution, light
yellow to yellow-green liquid (R. T. Vanderbilt Co.)

Hyamine 1622: Diisobutylphenoxyethoxyethyl dimethyl benzyl
ammonium chloride, white crystalline powder (Rohm and Hass
Co.)

Concentration

Figure C.1, Inputs and Concentrations Anticipated for
Fecal/Wash Water Loop Onboard a Six-Man Modular Space Sta-
tion, represents the cases 1 through 5C occurring in the
system. The concentrations of the active ingredient in all
these cases in deionized silver dosed water were as follows.

Case No.	Concentrations %	
	Detergent	Germicide
1	0.0598	0.0414
2	0.1198	0.0828
3	1.195	0.825
4A	0.1087	0.0751
4B	0.725	0.50
4C	2.179	1.50
5A	0.5859	0.4041
5B	3.95	2.73
5C	11.60	8.0

C.1.2 Test Procedure

First of all, the amounts of 100% active ingredients
were calculated for different cases. Since the amount of per-
cent solids was unknown for Miranol JEM, Neutrogena bar soap,
Neutrogena rainbath soap, Ivory bar soap, and Tegobetaine-C, the
percent solids was determined by evaporating to dryness known
amounts of samples. Then the actual amounts of the various de-
tergents and germicides required in cases 1 through 5C were cal-
culated.

To determine which ingredient may cause a problem, the re-
spective amounts of individual ingredients were mixed in a half
gallon bottle with one liter of silver dosed water. The solu-
tion was allowed to homogenize on the bench for a day, and after
that turbidity and pH readings were taken on these cases. Some
of the important observations that were made at this point were
solubility, miscibility, coloration, appearance in the bottle
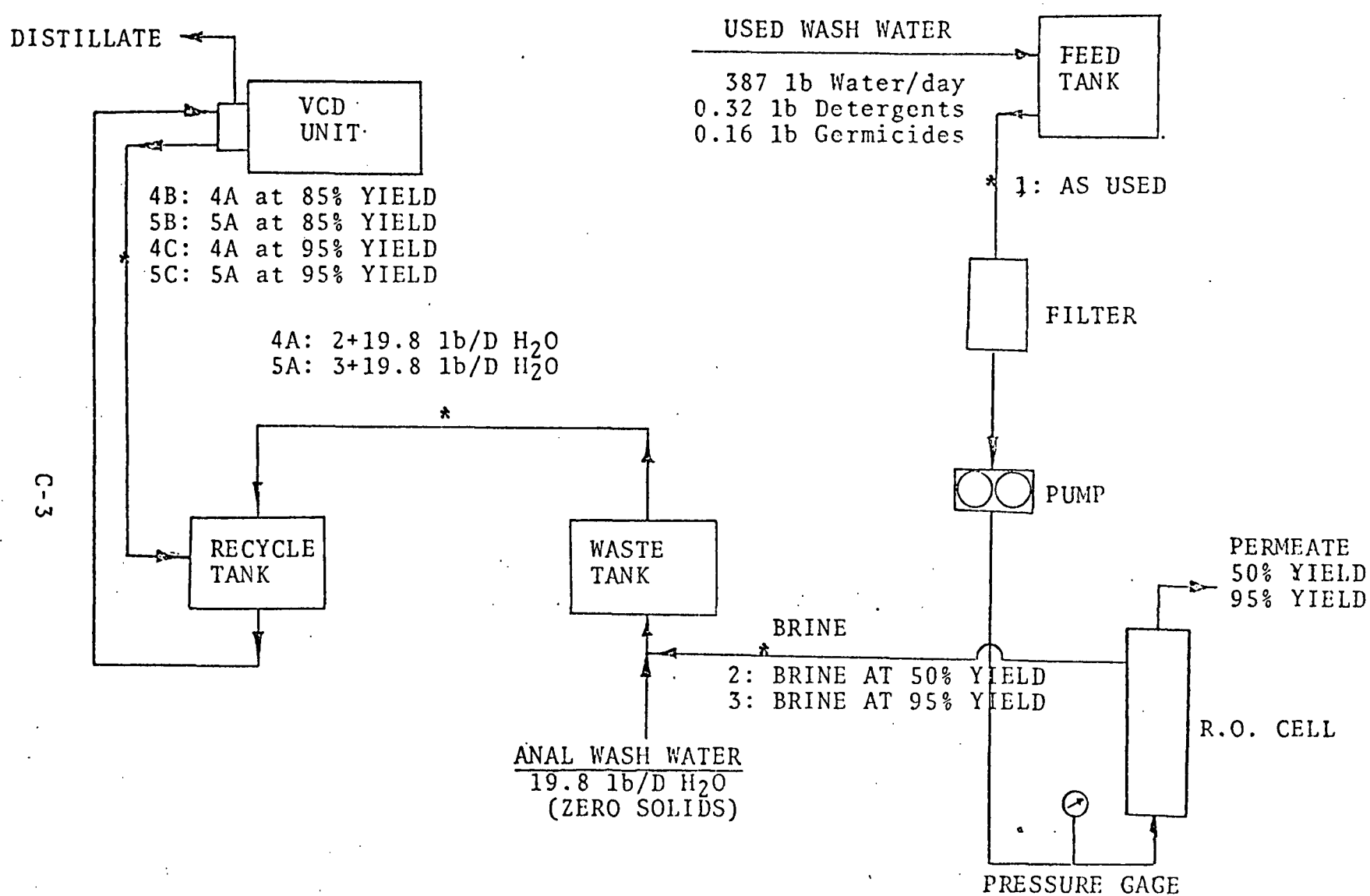and ease of filtration.

C H E M T R I C

Figure C-1 INPUTS AND CONCENTRATIONS ANTICIPATED FOR FECAL/WASH WATER LOOP
ONBOARD A SIX-MAN MODULAR SPACE STATION

To determine filtration required to reduce turbidity to less than 0.1 Jackson Units, the solution was filtered through membrane filters (Millipore) ranging in size from 100 microns to 50 millimicrons (14 steps). Any filter plugging problems were observed at this point. Corning pH Meter Model 7 was used for pH measurements. Hach Laboratory Turbidimeter Model 2100A was used for turbidity readings. Powders and liquids were weighed on Voland Model 220-D balance. The most promising individual ingredients were mixed in groups (detergent + germicide).

C.1.3 Test Results and Discussion

The results of the Static and Jar Tests performed are tabulated in Tables C-1 to C-17. Tables C-1 to C-3 present the results on the germicides, Tables C-4 to C-11 present the results on the detergents, and Tables C-12 to C-17 present results on combinations of detergents and germicides.

Liquid Vancide BN is the best germicide tested - Hyamine 1622 is second best and Vancide BN powder is the worst. From the tabulated results, it is apparent that the liquid Vancide BN does not have any solubility problem, plugging problem or higher turbidity like powdered Vancide BN. The JTU of 1 is readily obtained while very few additional filtration steps are required to achieve 0.1 JTU. Moreover, powdered Vancide BN contains undissolved residue which causes plugging problems. Hyamine 1622 requires additional filtration - i.e., more than liquid Vancide to achieve a JTU of 1 and 0.1. Other than some insoluble precipitate which appears in the Hyamine 1622, it does not have any other undesirable characteristics.

Among the three amphotheric surfactants, viz., Miranol C2M, Miranol JEM and Tegobetaine-C, Miranol C2M is the best while the latter two are about the same. Miranol C2M produces a water white solution in the first two cases and light yellow solution in case three; a JTU of 1 is readily obtained in the first two cases and it is easy to filter. Miranol JEM produces a whitish solution in cases with low concentrations; in case 4C, the color of the solution is white, and yellow-colored oil drops develop after 12 hours. Tegobetaine-C produces hazy-water colored solution; a JTU of 1 is not achieved even with a 50 millimicron filter in case three.

C H E M T R I C

Table C-1   HYAMINE 1622 IN SILVER DOSED WATER

Case No.	Hyamine* Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	0.419	2.2	5.7	.22 u	100 mμ	1 thru 5 C, Insoluble precipitate, probably iron and lint.
2	0.836	3.5	5.3	.45 μ	100 mμ	
3	8.34	4.6	4.85	.45 μ	100 mμ	
4A	0.758	---	5.6	---	---	
4B	5.06	---	5.1	---	---	
4C	15.16	---	4.55	---	---	
5A	4.09	---	5.2	---	---	
5B	27.58	---	4.35	---	---	
5C	80.8	---	4.2	---	---	

* Active Ingredient 99%

Table C-2   <u>POWDER VANCIDE BN IN SILVER DOSED WATER</u>

Case No.	Vancide* Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	0.450	29	8.0	100 mμ	50 mμ	Vancide is not completely dissolved, poor reproducibility or turbidity reading
2	0.899	39	8.25	100 mμ	50 mμ	Same as above
3	8.95	525	9.3	100 mμ	Not Achieved	JTU of 0.1 can not be achieved, and same as 1
4A	0.816	36	8.25	---	---	Same as 1
4B	5.43	500	9.1	---	---	Layer of undissolved Vancide is observed in the bottom of the jar
4C	16.30	570	9.6	---	---	Same as above and poor reproducibility of turbidity readings
5A	4.39	260	8.95	---	---	Same as 4B and turbidity reading not reproducible
5B	29.63	300	9.95	---	---	Same as 4C plus turbidity reading is low because of colored solution
5C	86.98	290	10.4	---	---	Same as above

* Active Ingredient 92%

Table C-3 <u>LIQUID VANCIDE BN IN SILVER DOSED WATER</u>

Case No.	Vancide* Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	1.190	0.8	9.4	None	0.22 μ	
2	2.390	0.5	10.0	None	5.0 μ	
3	23.95	1.2	11.35	14 u	1.2 μ	Pale yellow color develops after a day
4A	2.17	0.58	9.9	---	---	
4B	14.51	1.0	10.65	---	---	
4C	43.50	0.95	11.45	---	---	Low turbidity reading because of light yellow color of solution
5A	11.71	0.65	10.65	---	---	
5B	79.0	1.9	11.7	---	---	
5C	232.0	0.52	12.3	---	---	Very low turbidity reading is the effect of yellow color

* Active Ingredient 50%

Table C-4   MIRANOL C2M IN SILVER DOSED WATER

Case No.	Miranol* Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	1.495	0.98	8.85	None	50 mµ	Water white solution
2	2.989	0.8	8.9	None	50 mµ	Same as above
3	29.95	1.95	9.7	100 mµ	50 mµ	Light yellow solution
4A	2.719	2.0	9.0	---	---	Same as 1
4B	18.11	1.1	9.6	---	---	Same as 1
4C	54.4	1.8	9.85	---	---	Yellow color solution
5A	14.65	0.8	9.5	---	---	Same as 1
5B	98.7	3.2	9.9	---	---	Dark yellow solution
5C	290.0	3.4	9.95	---	---	Same as above

* Active Ingredient 40%

Table C-5   IVORY BAR SOAP IN SILVER DOSED WATER

Case No.	Ivory** Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	0.664	100	9.55	100 mμ	50 mμ	14 μ filter plugged gradually, and slow filtration rate at 100 mμ
2	1.330	100	9.60	100 mμ	50 mμ	Same as above and colloidal in appearance
3	13.25	*	10.3	*	*	* Paste-like appearance, impossible to take sample and soap is not completely soluble
4A	1.202	70	9.5	---	---	Appearance of solution is colloidal
4B	8.05	800	10.3	---	---	Jello-like solution
4C	24.19	---	10.30	---	---	Solution is like a paste of soap
5A	6.51	---	9.9	---	---	Same as 4B
5B	43.90	---	10.30	---	---	Same as 4C.
5C	128.9	---	10.30	---	---	Looks like a heavy paste

** Active Ingredient 90%

Table C-6 <u>NEUTROGENA BAR SOAP IN SILVER DOSED WATER</u>

Case No.	Neutrogena* Conc. (g/l)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	0.656	220	8.05	100 mμ	100 mμ	14 μ filter plugged gradually, and slow filtration rate at 100 mμ
2	1.311	380	8.4	100 mμ	50 mμ	Same as above
3	13.12	800	9.1	50 mμ	Not Achieved	14 μ filter plugged readily, and very slow filtration rate at 100 mμ
4A	1.191	280	8.4	---	---	
4B	7.96	820	9.1	---	---	Appearance of solution is colloidal and settleable
4C	23.90	480	9.1	---	---	Appearance of solution is colloidal, settleable and jello like
5A	6.44	800	9.1	---	---	Same as 4B
5B	43.40	---	9.15	---	---	Appearance of solution is like pearlescent
5C	127.10	---	9.15	---	---	Same as above

* Active Ingredient 91%

Table C-7   TEGOBETAINE-C IN SILVER DOSED WATER

Case No.	Tegobetaine-C* Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	1.498	17.0	6.05	1.2 μ	.45 μ	Hazy-water color, higher turbidity reading because of haziness
2	2.994	14.0	5.1	0.8 μ	100 mμ	Same as above
3	29.86	7.7	4.8	Not Achieved	Not Achieved	Water-white solution
4A	2.716	16.5	5.1	---	---	Same as case 1
4B	18.09	8.05	4.8	---	---	Slightly hazy water color
4C	53.81	8.0	4.8	---	---	Same as case 3
5A	14.64	7.1	4.8	---	---	Same as case 3
5B	100.1	8.3	4.75	---	---	Faint yellow tint
5C	284.0	6.2	4.8	---	---	Clear yellow color

* Active Ingredient 38.7%

Table C-8 MIRANOL JEM* IN SILVER DOSED WATER

Case No.	Miranol** Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	1.11	3.5	9.45	0.8 µ	100 mµ	Color of solution is water white
2	2.23	9.5	9.7	0.8 µ	100 mµ	Pale white color
3	22.3	380.0	10.35	0.22 µ	50 mµ	Whitish in color and remains so while passed through 0.22 µ filter
4A	2.03	---	9.65	---	---	Pale white color
4B	13.30	---	10.1	---	---	Whitish in color
4C	40.20	---	10.35	---	---	White color solution and yellow colored oil drops develop after 24 hours
5A	10.91	---	10.1	---	---	Same as 4B
5B	75.80	---	10.4	---	---	Pale yellow colored and yellow colored oil drops develop after 12 hours
5C	216.0	---	10.4	---	---	Yellow colored liquid - no oil drops

* Not used for personal hygiene

** Active Ingredient 37%

Table C-9   NEUTROGENA BAR SOAP* IN SILVER DOSED WATER

Case No.	Neutrogena** Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	0.203	35	7.75	100 mμ	100 mμ	14 μ filter plugs gradually and slow filtration rate at 100 mμ
2	0.409	120	8.0	100 mμ	100 mμ	Same as above and colloidal in appearance
3	4.06	850	9.1	100 mμ	50 mμ	14 μ filter plugs readily and slow filtration rate at 100 mμ
4A	0.370	---	8.0	---	---	Same as 2
4B	2.54	---	9.0	---	---	Same as 2
4C	7.54	---	9.1	---	---	Appearance of solution is colloidal and settleable
5A	1.99	---	8.6	---	---	Same as 2
5B	13.58	---	9.1	---	---	Appearance of solution is colloidal and settleable
5C	39.50	---	9.1	---	---	Appearance is like pearlescent

* Used for personal hygiene only

** Active Ingredient 91%

Table C-10   IVORY BAR SOAP* IN SILVER DOSED WATER

Case No.	Ivory** Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	0.207	52	8.7	100 mμ	100 mμ	14 μ filter plugged gradually, and slow filtration rate at 100 mμ
2	0.414	110	9.2	100 mμ	50 mμ	Same as above and colloidal in appearance
3	4.11	420	9.95	***	***	*** Jello-like solution not possible to filter through 14 μ filter
4A	0.374	---	9.2	---	---	Same as 2
4B	2.58	---	9.8	---	---	Same as 2
4C	7.66	---	10.1	---	---	Jello-like solution; not possible to read turbidity easily
5A	2.02	---	9.75	---	---	Same as 2
5B	13.70	---	10.15	---	---	Solution is like paste
5C	40.0	---	10.2	---	---	Same as above

*   Used for personal hygiene only

** Active Ingredient 90%

Table C-11   NEUTROGENA RAINBATH BODY SOAP GEL* IN SILVER DOSED WATER

Case No.	Neutrogena** Conc. (g/1)	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	0.565	8.0	5.30	0.22 μ	100 mμ	
2	1.138	8.3	5.40	0.22 μ	100 mμ	Whitish solution
3	11.30	14.0	6.8	100 mμ	50 mμ	Pale yellow solution
5C	110.0	8.0	7.6	---	---	Low turbidity reading is the effect of yellow color of solution

---

* Used for personal hygiene only

** Active Ingredient 32.7%

Table C-12   MIRANOL C2M + HYAMINE 1622 IN SILVER DOSED WATER

Case No.	Concentration (g/l) Miranol*	Hyamine**	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	1.495	0.419	1.4	8.45	0.8 μ	0.22 μ	Colorless solution
2	2.989	0.836	4.5	8.50	0.45 μ	50 mμ	Same as above
3	29.95	8.34	8.7	9.2	Not Achieved	Not Achieved	JTU of 1 cannot be achieved

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* Active Ingredient 40%

** Active Ingredient 99%

Table C-13 MIRANOL C2M + LIQUID VANCIDE BN IN SILVER DOSED WATER

Case No.	Concentration (g/l) Miranol*	Concentration (g/l) Vancide**	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	1.495	1.190	0.25	9.4	None	100 mμ	Colorless solution
2	2.989	2.390	0.24	9.7	None	100 mμ	Same as above
3	29.95	23.95	0.76	10.7	None	Not Achieved	Yellow-colored solution and JTU of 0.1 cannot be achieved

* Active Ingredient 40%

** Active Ingredient 50%

Table C-14   MIRANOL JEM + HYAMINE 1622 IN SILVER DOSED WATER

Case No.	Concentration (g/l) Miranol*	Hyamine**	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	1.615	0.419	0.34	9.5	None	100 mμ	Water-white solution
2	3.22	0.836	0.40	9.55	None	100 mμ	Same as above
3	32.2	8.34	120.0	9.9	Not Achieved	Not Achieved	Whitish solution and JTU of 1 cannot be achieved

* Active Ingredient 37%

** Active Ingredient 99%

C-18

Table C-15 MIRANOL JEM + LIQUID VANCIDE BN IN SILVER DOSED WATER

Case No.	Concentration (g/l) Miranol*	Vancide**	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	1.615	1.190	24.0	10.0	100 mµ	100 mu	Whitish solution
2	3.22	2.390	45.0	10.1	0.22 µ	100 mu	Same as above
3	32.2	23.95	130.0	10.85	50 mµ	50 mµ	Pale yellow solution with yellow oil drops

* Active Ingredient 37%

** Active Ingredient 50%

Table C-16   MIRANOL C2M + NEUTROGENA RAINBATH* + HYAMINE IN SILVER DOSED WATER

Case No.	Concentration (g/l) Miranol **	Neutrogena ***	Hyamine ****	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Req'd. For 0.1 JTU	Remarks
1	1.035	0.565	0.419	170	7.8	0.8 μ	0.45 μ	Whitish in color
2	2.06	1.138	0.836	250	8.3	0.22 μ	100 mμ	Same as above
3	20.6	11.30	8.34	90	9.1	Not Achieved	Not Achieved	Pale yellow jelly-like layer in the bottom. Readily plugs the 100 mμ filter
5C	200.0	110.0	80.8	40	9.2	---	---	Slightly thinner than honey-like, yellow-colored liquid

*   Used for personal hygiene only

**   Active Ingredient 40%

***   Active Ingredient 32.7%

**** Active Ingredient 99%

Table C-17   MIRANOL C2M + NEUTROGENA RAINBATH* + LIQUID VANCIDE BN IN SILVER DOSED WATER

Case No.	Concentration (g/l) Miranol **	Neutrogena ***	Vancide ****	Turbidity (JTU's)	pH	Filter Req'd. For 1 JTU	Filter Re 'd. For 0.1 JTU	Remarks
1	1.035	0.565	1.190	6.3	9.65	0.8 μ	0.45 μ	Water white
2	2.06	1.138	2.390	6.5	10.00	0.22 μ	100 mμ	Water white
3	20.6	11.30	23.95	1.25	11.0	Not Achieved	Not Achieved	Clear yellow - low turbidity due to color
5C	200.0	110.0	232.0	---	---	---	---	Yellowish-red solution

*   Used for personal hygiene only

**   Active Ingredient 40%

***   Active Ingredient 32.7%

****   Active Ingredient 50%

Of the mild personal skin cleaners tested, Neutrogena Rainbath is the best, whereas, bar soaps are not compatible in the system. The bar soaps plug a 14 micron filter readily. Their appearance in the solution is colloidal, gelatin and pearlescent, depending upon their concentration in the silver-dosed water. All these forms plug filters readily. In case 3 of Ivory soap, it was not even possible to obtain 30 ml of sample through a 100 millimicron filter. Neutrogena Rainbath Body Soap Gel does not have any apparent problem in filtration or appearance in the solution, therefore, it is a better skin cleaner for the application.

The test results presented in Tables C-12 to C-17 show the combinations of detergents and germicides. From the results in the first four tables (C-12, C-13, C-14 and C-15), it is apparent that Miranol C2M plus liquid Vancide BN in silver-dosed water is the best combination; JTU's of 1 or 0.1 are readily obtained with these agents. The next best combination is either Miranol C2M + Hyamine 1622 or Miranol JEM + Hyamine 1622. Miranol JEM produces yellow oil drops in case 3 in both groups. One reason why Miranol JEM + Hyamine 1622 is placed in the catagory of Miranol C2M + Hyamine 1622, is that a JTU of 1 is readily obtained with this combination in the first 2 cases. The combination of Miranol JEM + liquid Vancide BN required finer filtration to achieve a JTU of 1.

The last two tables (C-16 and C-17), present results with two detergents in combination with Germicides. Miranol C2M + Neutrogena Rainbath Body Soap Gel + liquid Vancide BN in silver-dosed water is better than Miranol C2M + Neutrogena Rainbath Body Soap Gel + Hyamine 1622. The main reason behind this is in case 3 where a pale yellow gelatin layer appears in the bottom of the jar and the solution readily plugs the 100 millimicron filter. The group with two detergents and liquid Vancide BN requires more prefiltration than Miranol C2M + liquid Vancide BN alone.

C.1.4   Conclusions and Recommendations

The following conclusions are based upon results of the experimental investigations conducted during the performance of the static (jar) tests.

A.   Liquid Vancide BN is the most promising germicide, while the Hyamine 1622 is second best.

B.   Powder Vancide BN is rejected on the basis of a plugging problem, because of insoluble residue in the formulation.

C.   On the basis of ease of filtrations, and miscibility prop-
      erty, Miranol C2M is the best amphotheric detergent. Mir-
      anol JEM forms oil drops after twelve hours and Tegobe-
      taine-C produces a hazy water solution.

D.   Miranol C2M and liquid Vancide BN is the best combination
      of detergent and germicide with respect to turbidity and
      filtration requisites.

E.   The bar soaps, Ivory and Neutrogena, contain suspended
      solids which readily plug filters. Their use would also
      develop problems in the distillation units because of
      solubility limits (ca 10%).

F.   Neutrogena Rainbath Body Soap Gel does not contain any
      suspended solids in it's formulation and therefore is a
      preferred skin cleanser for RO applications.

Since concern has been expressed by others regarding the
use of a detergent with biocidal properties (namely, Miranol),
Neutrogena Rainbath Soap Gel is recommended only for personal
hygiene (i.e., showering and face and hand washing). Miranol
C2M and Vancide BN would then be used only for clothes and
dishwashing. The combination of these three agents does not
present any significant filtration problems (second best group).
It is recommended that all cleansing agents be prefiltered be-
fore storing onboard in order to reduce inflight filtration re-
quirements.

C.2   Filtration Tests

Using the selected cleansing agents from the static (jar)
test, used wash waters from the shower, sinks, clothes washer
and dish washer were subjected to staged filtrations and tur-
bidity determinations. The objective was to establish the fil-
tration criteria.

C.2.1   Test Procedure

One-man batches of silver-dosed deionized water and
cleansing agents were prepared in accordance with the data list-
ed in Table C-18. Subsequently, these batches of water and
cleansing agents were used in showering, face and hand washing,
clothes washing and dish washing of one man, his clothes (cot-
ton knit shirt, work pants, socks and undergarments) and his
dishes (cup, plate, soup dish and silverware).

Table C-18   ONE MAN WASH WATER REQUIREMENTS

Washing Application	Amounts of Water (liters)	Amount of Ingredients (grams)		
		Neutrogena Rainbath*	Miranol C2M-SF**	Liquid Vancide BN***
Showering	3.628	11.10	0.0	0.0
Hands & Face	1.814	5.54	0.0	0.0
Clothes	16.629	0.0	20.8	16.61
Dish	6.804	0.0	8.38	6.71

*   Active ingredient 32.7%
** Active ingredient 40.0%
***Active ingredient 50.0%

The used (soiled) wash water was collected and allowed to homogenzie for 1/2 hour on the bench before turbidity and pH determinations were made.   The used wash water was filtered through membrane filters (Millipore) ranging in size from 100 microns to 50 millimicrons.   Corning pH Meter Model 7 was used for pH determinations and Hach Laboratory Turbidimeter Model 2100A was used for turbidity determinations.

C.2.2   Test Results & Discussion

The results of used wash water filtration tests and turbidity determinations are listed in Table C-19.   The tabulated information shows that used shower water is the most turbid, clothes wash water second, dish water third, and used face and hand wash water the least turbid.

The data further indicates that filtration down to 100 millimicrons will be required to achieve a turbidity of 1 JTU.   Turbidities of 0.1 JTU will never be practically achieved with membrane filtration; adjunct pretreatment (i.e., flocculation combined with coagulant aids) will probably be required to achieve this criterion.

C.3   Compatibility Tests

One (1) DuPont Permasep Permeator with a B-9 barrier and two (2) Westinghouse 4 x 4 Basic Modules were subjected to combination static storage/performance tests to determine the effects of selected cleansing agents on the respective RO membranes.   The following units were tested.

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Table C-19 USED WASH WATER TURBIDITY

Filter Size Microns	Turbidity After Filtration (Jackson Units)			
	Shower	Clothes	Dishes	Face & Hands
Original	130.0	72.0	60.0	50.0
104.0	130.0	72.0	27.0	50.0
74.0	130.0	72.0	27.0	50.0
65.0	130.0	72.0	24.0	50.0
43.0	84.0	72.0	22.0	50.0
14.0	76.0	62.0	12.0	40.0
8.0	72.0	61.0	6.6	33.0
5.0	66.0	61.0	6.2	28.0
3.0	57.0	58.0	5.9	26.0
1.2	45.0	47.0	5.5	22.0
0.8	36.0	36.0	5.2	19.0
0.45	31.0	26.0	4.1	11.0
0.22	15.0	13.0	2.4	2.8
0.10	0.53	0.9	0.7	0.3
0.05	0.12	0.53	0.3	0.1

C H E M T R I C

(1) DuPont Permasep Permeator Model XB-9S, Serial No. 1558-B, 5-1/2" OD x 4-1/2" ID x 23" long.

(2) Westinghouse 4 x 4 Basic Module, Type A, Serial No. 4-41, 4" OD x 52" long.

(3) Westinghouse 4 x 4 Basic Module, Type A, Serial No. 4-93, 4" OD x 52" long.

The DuPont unit utilizes an asymetric hollow fiber membrane configuration. The hollow fibers are made from a proprietary aromatic polyamide polymer; the open ends of the hollow fibers are potted with epoxy into a tube sheet. Water under pressure permeates through the hollow fiber membrane wall and leaves the permeator after flowing down the bore of the hollow fiber. The shell and end plates are made from 6063-T6 aluminum.

The Westinghouse units utilize a tubular membrane configuration. The tubular membranes are produced by drop-casting a special cellulose acetate base solution in parallel holes cored through a length of resin-bonded sand matrix. Each unit contains 18 tubular elements, approximately one-half inch in diameter. Water under pressure permeates through the membrane wall, then passes through the resin-bonded sand structure and is removed through side taps. The jacket which supports the sand matrix and the end flanges is made from 304 stainless steel.

C.3.1 Test Apparatus

The schematic presented in Figure C-2 illustrates the design of the test set-up. Two pumps were included in the hydraulic circuit; a hydraulically activated pump (Hills-Mc-Canna Model MA11-332-D) was used to feed the RO units and develop the operating pressure, and a centrifugal pump (Cole-Parmer Instrument Co., Model 7003) was used to develop fore pressure and overcome the pressure drop of two filters in series. AMF/CUNO's Model GH Auto-Klean Filter (ca 88μ) was selected for the coarse filter, and their Model 1M1-40069-01 Micro-Klean Filter with a 3μ in-depth filter cartrdige was selected for the fine filter.

The flow through the membrane of the RO units was controlled by regulating the brine bleed-off; a micrometer-type needle valve (Whitey Model 22RS4) was employed. A hydraulic accumulator was incorporated to alleviate pressure fluctuations. A pressure relief valve was included to protect the system should a malfunction occur.
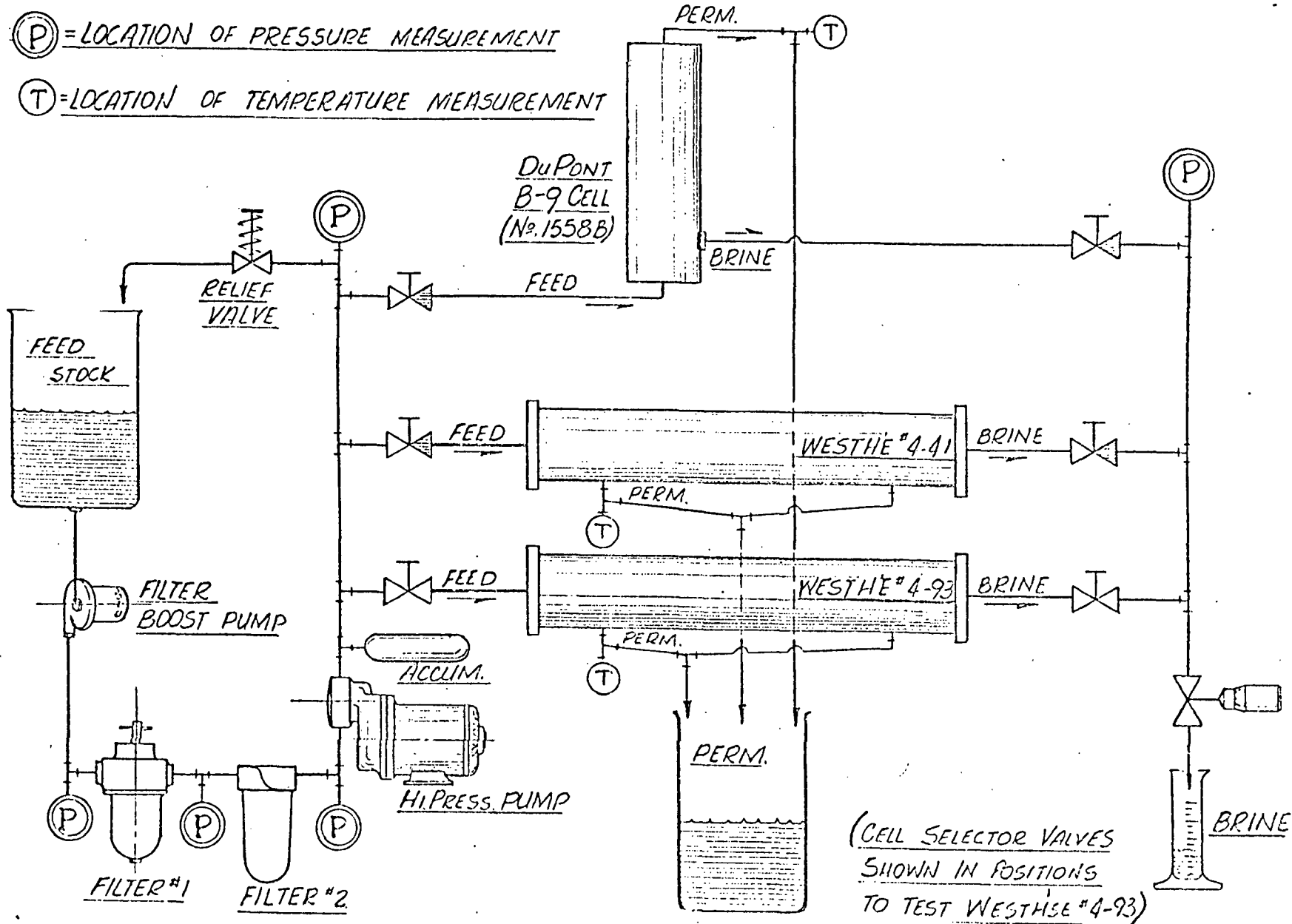
C H E M T R I C

(P) = LOCATION OF PRESSURE MEASUREMENT

(T) = LOCATION OF TEMPERATURE MEASUREMENT

DuPont
B-9 CELL
(N⁰.1558B)

PERM.

BRINE

FEED

FEED STOCK

RELIEF VALVE

FILTER BOOST PUMP

FEED   WESTHSE #4-41   BRINE

PERM.

ACCLIM.

FEED   WESTHSE #4-93   BRINE

PERM.

Hi. PRESS. PUMP

FILTER #1   FILTER #2

PERM.

(CELL SELECTOR VALVES SHOWN IN POSITIONS TO TEST WESTHSE #4-93)

BRINE

Figure C-2   FLOW SCHEMATIC FOR REVERSE OSMOSIS COMPATIBILITY TESTS

C.3.2 Test Procedure

Processing in a single pass (without recycle) and at a constant feed rate (ca. 10 l/hr) was selected as the operating condition for the compatibility test. Each unit was initially calibrated with silver-dosed deionized water to establish a baseline for subsequent comparisons.

After calibration, each unit (one a day) was fed test solution composed of cleansing agents for the entire day (8 hours). The feed pressure was determined at yields of 50%, 75%, 85% and 95%. After a day's run on one unit, the membrane of this unit was allowed to remain in contact with the resultant brine, soaking for at least three days. The permeate and brine recovered were analyzed for pH, turbidity, COD, and total solids. This procedure was then repeated again.

The test solution fed to the units was composed of the following:

(1)   41.3 g Neutrogena Rainbath Body Soap Gel
(2)   75.0 g Miranol C2M-SF
(3)   59.8 g Vancide BN liquid
(4)   72.48 l silver-dosed deionized water

The pH to Westinghouse Unit No. 4-93 was adjusted to 7.9 by adding 70 ml of 10% acetic acid to the above.

C.3.3 Results & Discussion

Compatibility tests were conducted over a 30 calendar day period. During this time, each unit was subject to a combination of static storage and performance tests. After the initial calibration with deionized water, each unit (one a day) processed the test solution for 8 hours in a cyclical pattern; usually, one day on and two days off. Each unit's membrane consequently was exposed to substances in the test solution (cleansing agents) for 27 days.

The results of the compatibility tests are shown in Figures C-3, C-4 and C-5 and in Table C-20. Figures C-3, C-4 and C-5 present the calibration data and permeate flow rates as a function of applied pressure for DuPont's Permasep Unit No. 1558-B and Westinghouse Units 4-41 and 4-93, respectively. Table C-20 lists and compares the operating characteristics and water quality of the three units at 95% recovery.
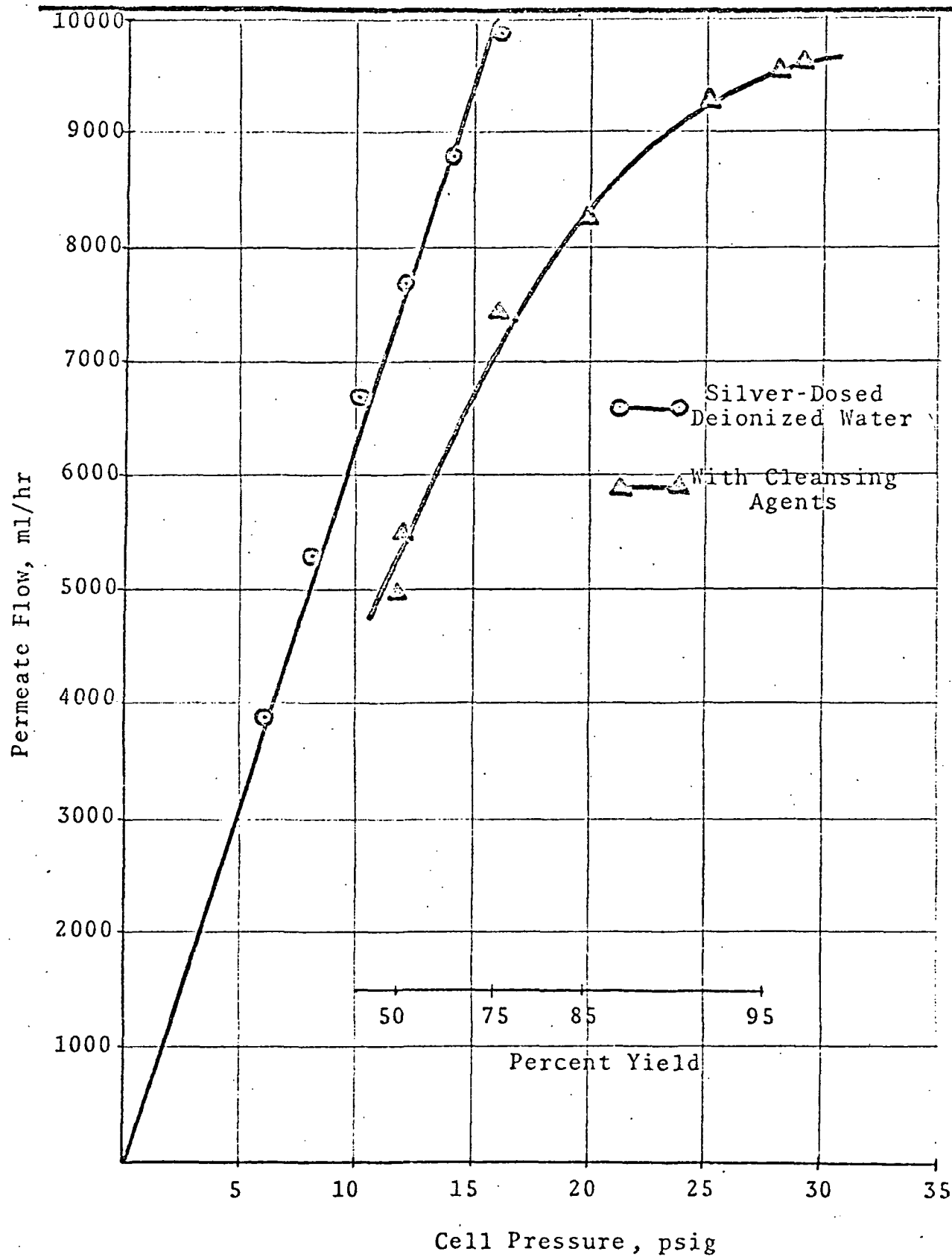
C H E M T R I C
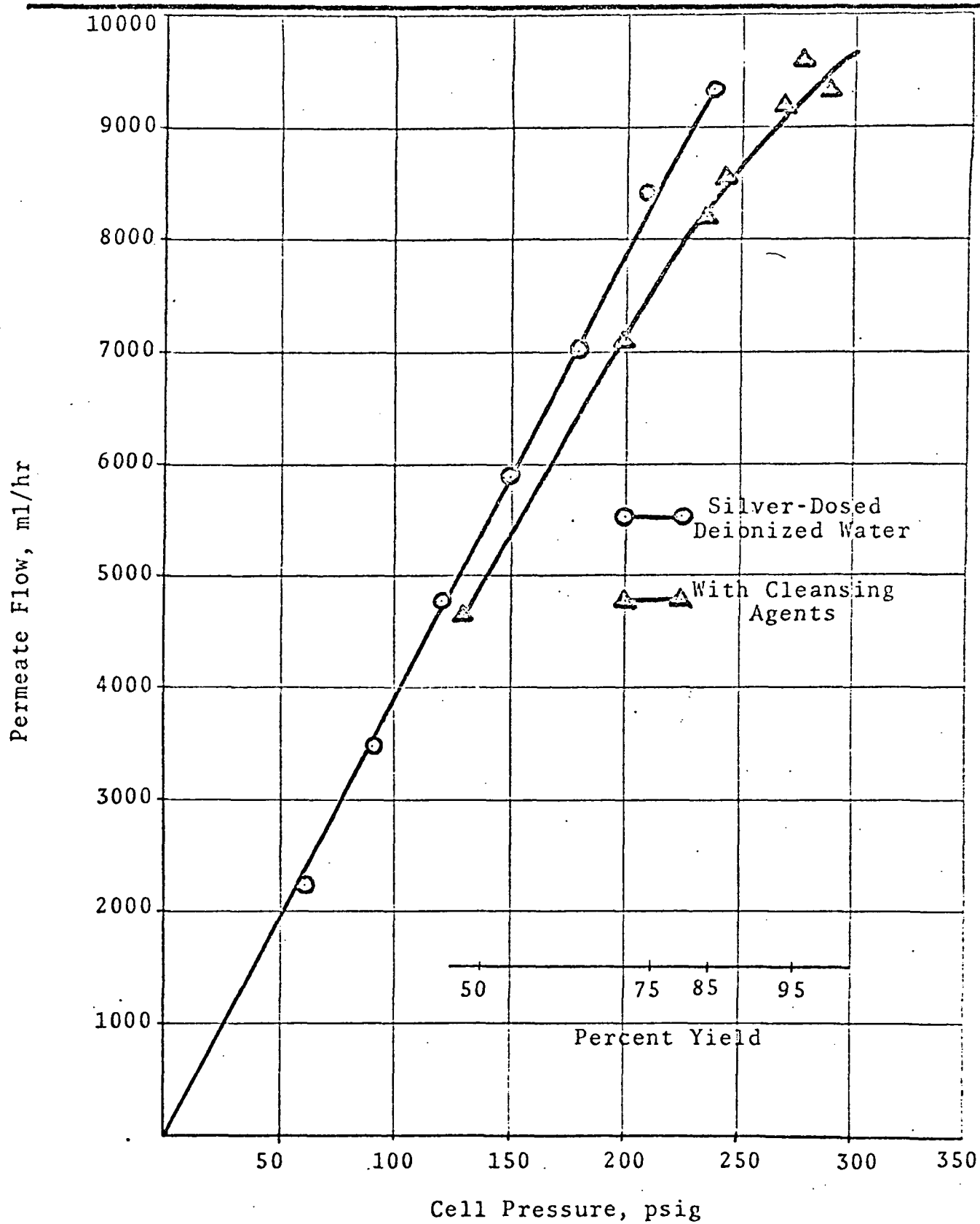
Figure C-3   CALIBRATED PERFORMANCE OF DU PONT PERMASEP NO. 1558-B·

C   H   E   M   T   R   I   C

Figure C-4   CALIBRATED PERFORMANCE OF WESTINGHOUSE UNIT NO. 4-41

C-30

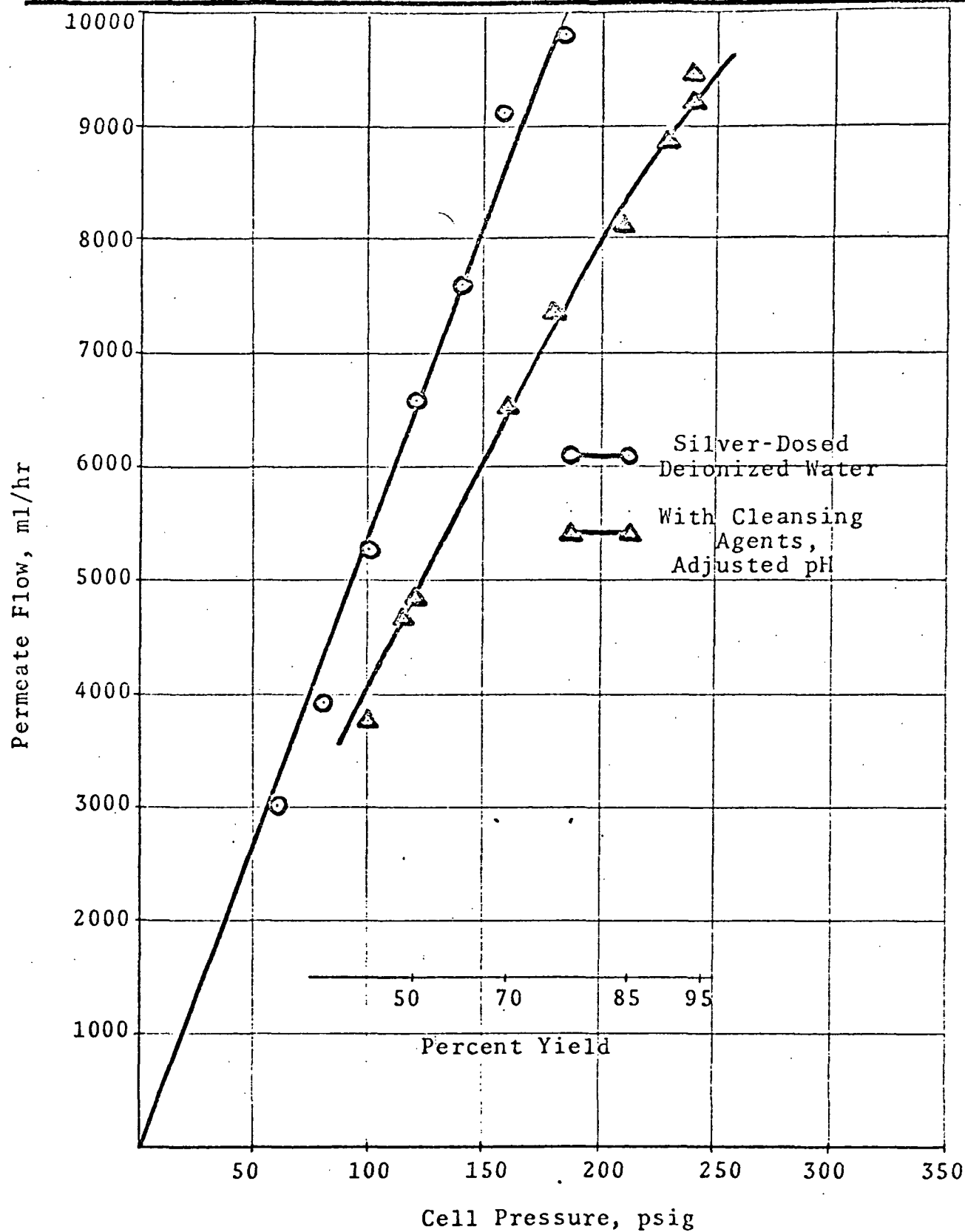C   H   E   M   T   R   I   C

Figure C-5   CALIBRATED PERFORMANCE OF WESTINGHOUSE UNIT NO. 4-93

C   H   E   M   T   R   I   C

Table C-20 OPERATING CHARACTERISTICS OF RO UNITS AT 95% RECOVERY

Unit Name & Number	Dupont 1558-B	Westinghouse 4-41	Westinghouse 4-93
Cell Pressure (psig)	25-29	270-290	230-240
Temperature (°F)	77-78	75-76	78-79
pH			
Feed	10.05-10.1	10.05-10.1	7.9
Brine	9.80-9.95	10.00-10.7	8.70-8.75
Permeate	9.85-10.2	9.35- 9.6	7.65-8.55
COD (ppm)			
Feed	1,256-1,850	1,260-1,460	1,300-1,600
Brine	26,680-32,000	16,320-26,000	9,000-19,000
Permeate	80-140	26-120	60-120
Turbidity (JTU's)			
Feed	0.3+0.05	0.3+0.05	110-118
Brine	6.5-8.3	0.64-0.94	30-32
Permeate	1.3-1.4	0.07-0.09	0.12-0.15
Total Solids (%)			
Feed	0.096-0.1027	0.094-0.101	0.102-0.105
Brine	1.002-1.121	0.509-1.299	0.812-1.156
Permeate	0.005-0.008	0.008-0.007	0.005-0.008
Rejection (%)	92.2-94.7	91.5-93.5	92.5-95.1

C H E M T R I C

From the tabulated results, it is apparent that all three units were producing permeate of excellent quality, averaging about 60 ppm total solids and 100 ppm COD. Figures C-3, C-4 and C-5 show that the DuPont unit was operated at relatively low pressures (30 psig) compared to the Westinghouse units (250-300 psig). The difference in the unit's permeability is attributed to differences in membrane area; the DuPont unit contains approximately 500 ft^2 of membrane area, whereas, the Westinghouse units contain approximately 10 ft^2 of membrane area.

DuPont Unit 1558-B and Westinghouse Unit 4-41 processed a feed stock with unadjusted pH (ca. 10.1). Westinghouse Unit 4-93 was operated with a pH adjusted to below 8 (ca. 7.9) according to the recommendations of Westinghouse.* No degradation in performance was observed with Westinghouse's Unit 4-41; however, it is well known that cellulose acetate's hydrolysis rate is a function of pH, increasing one order in magnitude for every pH unit. Adjustment of pH resulted in increased turbidity of the feed stock, see Table C-20. Acetic acid was selected over sulfuric, hydrochloric, nitric and citric acids because it produced the least turbidity; the other acids produced turbidities of 500-1000 JTU's.

C.3.4 Conclusions and Recommendations

Since no degradation in performance was observed with either the DuPont or Westinghouse unit while processing cleansing agents, it is concluded that both units were satisfactory for the application. A Westinghouse unit was recommended for the loop test on the basis of membrane configuration; the tubular configuration is capable of handling higher particulate loads than the fiber bundle used in DuPont's B9 unit.

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* Private Communications, John Koenst, Westinghouse R&D Center, Pittsburgh, Pennsylvania

C H E M T R I C